MAT-91-08

Optimization of a Cement Grout Formulation for Ducts Containing Post-Tensioned Cables

Ontario

Ministry of Transportation

Research and Development Branch

Ontario
Ministry of Transportation
Research and Development Branch

Documentation Page

MAT-91-08

Optimization of a Cement Grout Formulation for Ducts Containing Post-Tensioned Cables

Author(s): R. T. Hemmings and B. J. Cornelius
Matex Consultants, Inc.

Date Published: July, 1991

Published by: Research and Development Branch, MTO

Contact Person: Alan Ip, Materials
(416) 235- 4641

Abstract: Early stiffening reported in the field placement of a developmental, prepackaged cement (PPC) grout (comprising Type 10 Portland cement, silica fume, superplasticizer and aluminum powder) has been investigated. The thickening behaviour of the grout was duplicated in the laboratory, and its chemical, physical and rheological properties examined under conditions of mixing, pressure and temperature similar to those on site. The PPC grout exhibited complex rheological behaviour involving both a shear-dependent increase in viscosity (thixotropy) and time-dependent chemical thickening. Chemical incompatibility of the Portland cement and superplasticizer was subsequently identified as the cause of the stiffening, although the underlying mechanism remains to be determined.

Based on these findings, an optimized grout formulation was developed using alternative sources of Type 10 Portland cement and superplasticizer. The optimized grout was strongly cohesive, while having superior flow characteristics to the PPC material. Also, corrosion parameters related to durability were comparable or improved. It has been recommended that the optimized grout be tested at larger scale and under simulated field conditions to verify its acceptability. It will also be important to identify the nature of the superplasticizer-cement incompatibility, with the purpose of developing a practical cement specification for use in grouting operations.

Comments: Final Report of Research and Development Branch Project No. 22192, carried out under the Ontario Joint Transportation Research Program

Key Words: cement, grouts, grouting, admixtures, rheology, post-tensioned structures, prestressing steel, corrosion protection

Copyright Status: Crown copyright © 1991 Ministry of Transportation

MAT-91-08

Optimization of a Cement Grout Formulation for Ducts Containing Post-Tensioned Cables

R. T. Hemmings

B. J. Cornelius

Matex Consultants Inc.

92-00616/

Published by
The Research and Development Branch
Ontario Ministry of Transportation

Published without prejudice as to the application of the findings.
Crown copyright reserved; however, this document may be reproduced for non-commercial purposes with attribution to the Ministry.

For additional copies, contact:
The Editor, Technical Publications
Room 320, Central Building
1201 Wilson Avenue
Downsview, Ontario
Canada M3M 1J8

Telephone: (416) 235-3480
Fax: (416) 235-4872

July 1991

Table of Contents

Aknowledgements v

1/ Introduction 1

1.1/ Background 1

1.2/ Objectives 2

2/ Field Observations 3

3/ Investigation of Physical and Chemical Characteristics of New Grout 5

3.1/ Materials 6

3.2/ Mixing Method 7

3.3 Physical Properties 7

3.4/ Rheological Properites 8

3.4.1/ Qualitative Assessment 8

3.4.2/ Semi-Qualitative Measurement of Viscosity 9

3.4.3/ Dynamic Mechanical Analysis 10

3.4.4/ Effect of Pressure on Flow and Bleeding 11

3.5/ Chemical Properties 12

3.6/ Summary of Properties 14

4/ Screening Tests to Modify Grout Formulation 16

4.1/ Design Criteria 16

4.2/ Materials 17

4.3/ Optimization Trials 18

4.3.1/ Effect of Cement 18

4.3.2/ Effect of Silica Fume and Aluminum Powder 19

4.3.3/ Effect of Superplasticizer 20

4.3.4/ Temperature and Pressure Effects 21

4.3.5/ Selection of Optimum Formulation 21

5/ Testing of Modifed Formulation 22

5.1/ Physical Properties 22

5.2/ Rheological Properties 22

5.3/ Durability Related Properties 23

6/ Conclusions and Recommendations 24

References 27

List of Tables and Figures

Tables

1/ Target specifications for post-tensioning grout 28

2/ Comparison of physical and chemical properties of reference grouts 28

3/ Bound water and calcuim hydroxide contents of hydrating PPC grout 29

4/ Chemical and physical properties of portland cements 29

5/ Mix formulations for grout screening tests 30

6/ Mix formulations for rheological screening tests 30

7/ Effect of cement type on the setting properties of grout trial mixes (SMF superplasticizer) 31

8/ Effect of cement type on the rheological properties of grout trial mixes (SMF superplasticizer) 31

9/ Effect of silica fume on the setting properties of grout trial mixes (Cement A 10) 31

10/ Effect of silica fume on the rheological properties of grout trial mixes (Cement A10) 32

11/ Effect of aluminum powder on the setting properties of grout trial mixes (Cement A10/SMF Superplasticizer) 32

12/ Effect of aluminum powder type on the rheological properties of grout trial mixes (Cement A10/SMF Superplasticizer) 32

13/ Effect of superplasticizer type on the setting properties of grout trial mixes (Cement A10) 33

14/ Effect of superplasticizer type on the rheological properties of grout trial mixes (Cement A10) 33

15/ Effect of temperature on the efflux rate of the PPC and optimized (B10-SP3) grout mixes 33

16/ Summary of physical and chemical properties of optimized grout 34

Figures

1/ Bridge construction site on Highway 401, near Woodstock, Ontario, (*upper*) and view of longitudinal tendons prior to grouting (*lower*) 35

2/ High-shear mixer and grout pump 36

3/ Filling transverse tendon ducts with grout 37

4/ Sealing off plastic riser tubes 37

5/ Mixers used for preparation of cement grouts: (a) Hobart mixer for blending (2 min); and (b) Cowles Model 1VG for high-shear mixing (15 sec) 38

List of Tables and Figures (continued)

Figures

6/ Thixotropic behaviour of PPC grout 39

7/ Measurement of relative viscosity using a Brookfield RVT viscometer 40

8/ Variation of relative viscosity vs. time at 22°C for (a) control PPC grout and (b) PPC grout 41

9/ Variation of relative viscosity vs. time for (a) PPC grout at 30° C and (b) PPC grout at 22°C 41

10/ Rheometrics RDA-700 mechanical spectrometer used for dynamic mechanical analysis of grout 42

11/ Strain sweep of PPC grout at 23°C (start of run 7 min after mixing) showing yield point at ~60% strain 42

12/ Time sweeps of PPC grout at 23°C at 1% strain (*upper plot*) and 20% strain (*lower plot*) (start of run, 5 min from time of mixing) 43

13/ Time sweeps of PPC grout at 35°C and 20% strain (8 min after mixing) 44

14/ Filter press (NL Varaoid/NL Industries) used to measure pressure induced fluid loss (i.e., water retentivity) in grouts 44

15/ Cell used for measurement of efflux rate of grout under pressure 45

16/ XRD (*upper*) and TGA (*lower*) traces for as-received (unhydrated) PPC grout 46

17/ XRD patterns showing progressive hydration of PPC grout during 1-hour period following mixing 47

18/ TGA curves showing progressive hydration of PPC grout during 1-hour period following mixing, with reference to the unhydrated and mature grout. Phases indentifed as follows: E, ettringite; CH, calcium hydroxide; C, calcium carbonate 48

19/ Effect of cement type on the relative viscosity of grout mixtures 49

20/ Effect of aluminum powder on the relative viscosity of grout mixtures 49

21/ Effect of superplasticizer type on the relative viscosity of grout mixtures 50

22/ Effect of temperature on the relative viscosity of the PPC grout and the B10-SP3 grout 50

23/ Compressive strength development of the PPC grout and the optimized grout 51

24/ Comparison of the electrical resistivity of the PPC grout and the optimized grout 51

Acknowledgements

The authors wish to acknowledge the Ontario Ministry of Transportation, Research and Development Branch for the funding and support of this study, which was conducted under MTO 88010/File 22192 at the Ontario Research Foundation (now Ortech International).

The authors also thank Mr. John Deans, C.E.T., Civil Research Department, Research Division, Ontario Hydro, Mr. Noel Mailvaganam of the Institute for Research in Construction, National Research Council, and Dr. Edwin Berry of Matex Consultants for their helpful discussions and insights. Mr. Rob Collins and Mr. Greg Murawsky at Ortech are also thanked for their careful experimental work. The cooperation of the following companies in providing sample materials for the study is gratefully acknowledged: SKW Canada, St. Marys Cement Co., St. Lawrence Cement, Lafarge Canada Inc., Lake Ontario Cement Limited, Mulco Chemicals, and Sternson Ltd.

1/ Introduction

1.1/ Background

The injection of a cementitious grout into the void surrounding the prestressing steel tendons and ducts in post-tensioned concrete serves two purposes: (a) to protect the prestressing steel from deterioration due to the influences of corrosion during service; and (b) in some instances, to provide a bond between the prestressing steel and the concrete members in order to increase the ultimate resistance of the concrete member under load. The extent of corrosion protection and the degree of bond formation are both strongly dependent on the material characteristics of the grout.

To be effective, cementitious grouts for post-tensioned concrete should exhibit a number of desirable characteristics [1,2]:

- they should have a minimum water to cement (w/c) ratio to provide a high strength paste with low porosity and permeability;
- their viscosity must be such that they can be pumped into the tendon duct to completely fill all the voids surrounding the prestressing steel without bleeding or segregation;
- they should be expansive and not shrink in the plastic state or on hardening;
- they must set and harden to the required strength within a specified time period;
- they should not introduce aggressive chemical agents (e.g. chloride or nitrate ions) that may promote the corrosion of the steel or degrade the surrounding concrete.

Some target specifications for this type of grout are given in Table 1.

The Ontario Ministry of Transportation (MTO) has conducted investigations to improve existing grout mixtures and has developed a new formulation reported recently by Hope and Ip [3,4]. The most promising formulation developed by these workers was in the form of a preblended, prepackaged product for immediate on-site mixing, and typically consisted of the following combination of components:

- Type 10 Portland cement
- Silica fume (10–15% replacement of cement)
- Sulphonated melamine-formaldehyde condensates (SMF) superplasticizer (1% by mass of cement)
- Aluminum powder (uncoated, 0.0067%)

In this formulation, it is intended that the components should be mixed at an optimum water to cementitious materials ratio (w/c) of 0.33. This is a relatively low w/c and is desirable in a post-tensioning grout in terms of generating a low porosity paste which, in turn, can be expected to provide high strength, low chloride permeability and good protection against corrosion. The silica fume used in the formulation is a highly reactive pozzolan (supplementary cement) which further reduces porosity in the paste through formation of additional calcium silicate hydrate (C–S–H) gel, a process which results in the net consumption of much of the calcium hydroxide formed during the hydration of the Portland cement. The SMF superplasticizer is a surface-active chemical admixture which is necessary to achieve the low w/c and pumpability of the grout mix; it is essential for systems containing silica fume (which has a substantial water demand as a result of its high surface area). The aluminum powder is used to control shrinkage of the paste through generation of hydrogen gas as a result of its reaction with the alkalis formed during hydration of the cement. An uncoated aluminum powder will react more rapidly than the coated variety, given comparable particle size distributions.

Although the new grout formulation apparently functioned satisfactorily in the laboratory [3,4], it has behaved erratically under field conditions [5]. In particular, it has been reported that control of the quantity of mixing water has been difficult and early stiffening in the mix has been encountered. This has led to considerable difficulties with pumping of the mixture and has necessitated the undesirable measure of increasing the w/c ratio by adding water to promote flow. This behaviour, which appears to be manifested largely as a rheological problem, especially in relation to pumpability and the time available for injection after mixing, has given MTO cause for concerns relating to the quality control of the operation.

1.2/ Objectives

The present investigation was conducted to address the problems encountered with the new grout in the field. The principal objectives of the study are summarized as follows:

(i) To identify the nature of the rheological problems with the new grout;

(ii) To conduct laboratory screening experiments to develop a modified formulation which would make the new grout acceptable for field applications;

(iii) To conduct laboratory testing of a modified grout formulation for acceptability in terms of key parameters such as strength, electrical resistivity, chloride permeability and corrosion susceptibility.

This report summarizes the findings of the investigation, being organized into the following sections: observations made in the field to identify critical temperature, time and pressure parameters during the various phases of the grouting operation (Section 2); rheological and mechanistic examination of the new grout formulation (Section 3); laboratory trials to modify the new formulation for improved rheological performance (Section 4); and compliance testing of the modified grout formulation (Section 5). A final section presents the conclusions of the study and recommendations for additional research and development needs for cementitious grouts.

2/ Field Observations

Since the reported difficulties with the new grout formulation could manifest themselves in a number of ways, it was considered important to observe an actual tendon duct grouting operation in the field. This served both to familiarize the investigators with the equipment and procedures used to inject the grout into the tendon ducts and to allow the collection of critical temperature, time, and pressure parameters during the various phases of the operation. Among the critical parameters are: (a) the time of mixing, (b) the temperature rise during mixing, (c) the pressure used for injection and the likely hydraulic pressure heads encountered, (d) the time taken for injection, and (e) the temperature rise in the grout during injection.

Field observations during a grouting operation were conducted at a bridge construction site on Highway 401, near Woodstock, Ontario, on July 7, 1988 (Fig. 1). Grouting of the ducts for the transverse tendons of the bridge was being carried out at this time using the standard multi-component MTO Portland cement grout.*

An ambient temperature of 32-36°C had been experienced for several days prior to the date of grouting, and the temperature had reached ~34°C by 9:00 a.m. on the day of the inspection. Although these are certainly not considered to be typical or desirable conditions, they served the very useful purpose of defining the probable upper temperature limits which may be experienced in a grouting operation. This was thought to be important since there is likely a strong temperature dependency in the thickening, and hence pumpability, of the developmental grout.

*The conventional grout formulation used by MTO typically consists of the following components: CSA Type 30 Portland cement, non-chloride chemical water reducing agent (~0.28% mass of cement), and aluminum powder (~0.005% mass of cement). The maximum w/c ratio is specified as 0.50 for cables <45 m long and 0.55 for cables >45 m.

The grouting procedure was observed as follows. Since the standard MTO grout is multi-component, the uncoated aluminum powder and water-reducing agent components were first mixed on site in a plastic drum and homogenized by rolling the barrel along the ground. A measure of this admixture combination was added to the high-shear mixer (Fig. 2) along with four bags of Portland cement. Water was added by hand using a 5-gallon plastic pail until a certain visual consistency was obtained. The water arrived on site in a water truck, and was at a temperature of ~13°C at 9:45 a.m.

The temperature of the grout mixture was monitored in the mixer for ~15 min. After 2 min mixing, the temperature of the mixed grout was ~26°C, and increased to ~31°C within 8 min, at which time it remained reasonably constant. Pumping of the grout occurred 8–12 min after initial mixing at an indicated pressure of 0.55 MPa. When examined on the bridge deck during the actual injection of the transverse tendon ducts, the temperature of the grout was 32°C, before entering the duct, and 33°C upon exiting the duct. The total time required for a single tendon duct to be filled was ~2 min, which was generally from 10–15 min after initial mixing of the grout. There appeared to be some difficulty with sealing the plastic riser tubes emerging from the tendon ducts (Fig. 3) once the duct was filled with grout. The sealing was carried out with a large mechanical clamp device followed by manually tying off with twisted steel wire (Fig. 4). Leakage was observed on several of the seals which gives cause for concern that the seal was not gas tight and would, therefore, reduce the efficiency of the desired expansion caused by the hydrogen evolution from the reacting aluminum powder. On-site control tests by MTO showed that the grout had an unconfined expansion of 6–8% after ~30 mins [6].

It was evident from the field observations that the main temperature increase experienced by the grout occurred during the initial mixing procedure. The grout was only slightly warmer upon reaching the bridge deck after being pumped at 0.55 MPa, and increased in temperature by only 1°C between entering and leaving the tendon duct. This indicates that the temperature rise in the grout is by either the mechanical action of the high-shear mixer, or the exothermic cementitious hydration reactions of the cement, or, and more likely, a combination of both. There does not seem to be a significant temperature rise in the grout during its passage through the delivery pipe or the tendon duct.

These data were used as a guide to defining conditions for the laboratory examination of the new grout mixture.

3/ Investigation of Physical and Chemical Characteristics of New Grout

On reviewing the experimental grout formulation developed by Hope and Ip [3,4], it is possible to propose a number of physical or chemical factors, each with the potential to contribute to the rheological problems observed in the field with the fresh, unhardened grout:

(a) The SMF superplasticizer may be incompatible with other constituents in the system or may otherwise require modification to improve pumpability or increase the "pot-life" of the grout. The superplasticizer may also be sensitive to temperature, this being most significant during the grouting operation in which the temperature often exceeds 25–30°C.

(b) The effect of pressure on the rheological properties of the grout is unknown. Since the delivery pressures in a grout pump may exceed 0.55 MPa, the effect of pressure on the grout is an important consideration. This may be especially so for grouts which generate gas or which have varying thixotropic* tendencies.

(c) The silica fume may be reacting at an accelerated rate, again being sensitive to temperatures above 25–30°C.

(d) The Portland cement used may not be optimal. It may be too fast setting or contain enough soluble SO_3 to induce excessive sulphoaluminate precipitation at early ages. It may be high in C_3A and thus be incompatible with the superplasticizer.

(e) The use of aluminum powder may produce a flocculating alumina gel or contribute to sulphoaluminate precipitation. The uncoated aluminum powder may react too rapidly at elevated temperatures, leading to excessive gas generation and bloating-thickening of the mix.

(f) Too much air may be entrained into the grout using the field mixing techniques, thereby affecting the flow properties of the grout.

---

*Thixotropic fluids possess a structure, the breakdown of which is a function of time as well as shear rate. As the structure breaks down with constant shear rate, shear stress decreases. This structure can rebuild itself if not prevented from doing so by externally applied forces.

(g) In general, the energy input to the system (and hence the potential temperature rise and floc-breaking ability) during the mixing of the grout is not well understood. The energy input in the field grout pump is likely very different from that produced in the laboratory mixer. The shear energy, in particular, is likely an important factor in determining the pumpability of a thixotropic grout.

The first stage of the investigation was concerned with determining the physical and chemical characteristics of the new grout formulation with a view to establishing the nature of its rheological problems observed in the field and identifying which thickening mechanism was most likely operative. Test work carried out in the first stage was as follows: determination of basic physical properties; examination of temperature- and time-dependent rheological properties; and a limited mechanistic investigation of the chemical and mineralogical changes occurring during the early hydration of the grout. At the same time, this work served to identify the most appropriate test protocols which could be used subsequently to screen mixes with modified formulation (Section 4).

3.1/ Materials

Samples of the prepackaged grout mixture, originating from a commercial supplier, were provided by MTO from the same shipment which was tested in the earlier, unsuccessful MTO field trials [5]. As received, the grout powder appeared to be well packaged in 25 kg, 3-ply paper bags with a polyliner and showed no obvious signs of deterioration. It is understood that the prepackaged grout had been blended by the supplier according to the new formulation materials as follows:

Material	% of Total Mix (by mass)
Portland cement, Type 10	89.10301
Silica fume	9.90033
Superplasticizer, SMF type	0.99003
Aluminum powder, uncoated pharmaceutical grade	0.00663

This prepackaged cement grout mixture (referred to subsequently in this report as "PPC grout") was used as received, with no attempt being made to confirm that it was correctly proportioned.

For reference purposes, the component materials used in the PPC grout were obtained from the original suppliers (an equivalent grade of the aluminum powder was obtained from an alternative source) and blended to the intended proportions in the laboratory. This mixture is referred to as "Control PPC grout."

Trials were carried out on the as-received PPC grout with comparisons being made, as appropriate, with the Control PPC grout.

3.2/ Mixing Method

In general, the methods used for mixing laboratory mortars and concretes involve relatively low energy, or low-shear, mixing, such as might be achieved with a pan or Hobart-type mixer. These are adequate for most purposes and give good simulation of field mixing. However, in the case of a cement paste, and cement grouts in particular, low-shear mixing is very inefficient, leaving a large proportion of the particles as poorly dispersed agglomerates. For this reason, field grout mixers are of the high-shear variety (Fig. 2) in order to achieve the best possible mixing in the shortest time. While the high energy input from mixers of this type is efficient at breaking the floc structure of a hydrating cement, the shearing action also provides considerable heat energy to the system. These conditions can be expected to affect both the rate of hydration of the cement and the flow properties of the grout.

In evaluating grouts, therefore, the laboratory mixing method is far more critical than for mortars and concretes. This may be especially true for grouts which have shear-dependent (thixotropic) flow properties (see later). Although the shear energy input provided by the field grout mixer was not known, it was, nonetheless, considered important in this study to use high-shear rather than low-shear mixing. For this purpose, a non-standard mixing procedure was adopted, as follows: (a) the grout powder was hand-mixed with the gauging water (w/c = 0.33) and allowed to stand for 30 sec; (b) next, the materials were blended in a Hobart mixer for 1.5 min; and (c) finally, the blend was mixed for 15 sec using a Cowles Model 1VG high-shear mixer (Fig. 5). Unless otherwise stated, room temperature (~22°C) water was used for all mixing and, in some of the mixes, the temperature rise during mixing was also recorded. Some smaller trial mixes were prepared with a Dispermat™ high-shear mixer.

3.3/ Physical Properties

For comparison with the earlier work, the basic physical properties of the PPC grout mixture were examined, as follows: fluidity by flow cone (CAN3-A23.2-1B); consistency by cone penetrometer (ASTM C 472); slump by patty spread (ASTM C 230, modified); bleeding and expansion (ASTM C 940); time of setting (modified Vicat, ASTM C 807); and compressive strength development after curing for 1, 3, 7, 14, and 28 days (50 mm cubes, restrained, ASTM C 942).

The physical properties determined for the PPC grout are given in Table 2. The strength development was very good (reaching 34.0 MPa at 3 days, and 47.8 MPa at 28 days); there was no obvious bleeding, and the plastic expansion value of 9% fell in the required range of 8±2%. The flow tests (flow cone, consistency and slump) were carried out on fresh grout within 5 min of mixing. The flow time was ~55 seconds, although there was uncertainty in determining the end of the flow period due to the adherence of the grout to the walls of the flow cone (see discussion in Section 3.4). The consistency and slump did not appear unusual.

While these results are generally similar to those reported by Hope and Ip [3,4], two differences are apparent: (a) the flow time for the PPC grout is considerably longer than the 13.9 second flow time reported for a similar formulation (SF-B1) in the Queen's study; and (b) the compressive strengths for the PPC grout are ~80% of those in the Queen's study. The strength differences are thought to be porosity-related as a result of different rates of gas generation from the reaction of the aluminum, or air-entrainment during mixing. On this point, it should be noted that the Queen's work [3] involved low-shear mixing (ASTM C 938-80), whereas the more preferable high-shear mixing was used in this study. As noted above, high-shear mixing may promote more rapid reaction of the cement, which in turn will aggravate any tendency to thicken that exists.

3.4/ Rheological Properties

The rheological behaviour of the new grout formulation was examined in three ways: (a) qualitatively, to determine flow characteristics, (b) semi-quantitatively, to determine relative viscosity, and (c) quantitatively, by dynamic mechanical analysis. Work was also carried out to simulate the temperature and pressure conditions experienced by the grout during the various phases of mixing and injection.

3.4.1/ Qualitative Assessment

A sample of the PPC grout powder was gauged with water and examined visually in order to qualitatively assess changes in the flow behaviour of the system. The powder was mixed as described in Section 3.2, and the behaviour of the grout mixture observed at room temperature for ~1 hour.

Shortly after mixing, the grout mixture was quite fluid while being agitated gently by hand. However, almost immediately upon stopping the agitation the grout became quite stiff; i.e., the container could be held at 90° to the vertical and the material did not flow (Fig. 6). Gently tapping the side of the mixing container caused the grout to flow reasonably freely. After 30 minutes the mixture behaved in a manner similar to the freshly-mixed material, being easily fluidized with minimum effort. At 40 min, the mixture was noticeably thicker and required more effort for flow to be induced.

After 1 hour, considerable effort was required to induce flow in the mixture, and at this point it was considered unlikely that the grout would be pumpable by normal means. Similar results were obtained with the Control PPC grout mixture.

These observations provide an early indication that the PPC grout has thixotropic (pseudoplastic) tendencies. When the hydrating mixture is free of external stress it tends to form a weakly bound structure which, at early ages, is easily disrupted by modest external forces. The underlying cause of this behaviour may be either chemical or physical in nature, or a combination of both, as discussed below.

A second sample of the PPC grout was prepared using ice water and the resulting mixture chilled in ice and observed for its tendency to thicken. In striking contrast to the room temperature sample, the cold grout had no observable thixotropy and no tendency to solidify over the more than 4 hours during which it was observed.

3.4.2/ Semi-Quantitative Measurement of Viscosity

Semi-quantitative measurement of the change in viscosity with time were performed with a Brookfield RVT Viscometer and #C T-Bar spindle helical configuration, in conjunction with a Brookfield Helipath Stand (Fig. 7). A 1-litre polyethylene beaker was filled with 900 ml of the grout mixture to be evaluated and the T-Bar spindle was submerged ~1 cm below the sample surface in the centre of the beaker. At ~6 min after initial mixing, the viscometer was engaged at 10 rpm and the helipath stand started. The first dial scale deflection reading was taken at 6.5 min, with subsequent readings at 30-sec intervals to a total of 6 readings as the spindle transversed down through the sample. After the sixth reading, the spindle was raised and repositioned in another area in the centre of the sample (referred to as recycling) and another six readings were obtained. This was repeated twice more to obtain a total of 24 readings over a period of ~11.5 min. Scale deflection values were then converted to relative viscosity values in units of centipoise (cps),* based on an instrumental conversion factor of 1000 x for the 10 rpm speed with the #C spindle.

---

* The engineering unit of viscosity, the poise, is equivalent to 0.1 pascal seconds, Pa.S (and 1 cps = 1 Pa.S). Since the effective shear rate during measurements with the Brookfield RVT viscometer is not known, the viscosity data are only relative and are not directly comparable with those obtained by dynamic mechanical analysis (Section 3.4.3).

A plot of relative viscosity vs. time for the PPC grout is shown in Fig. 8. It can be seen that the relative viscosity of the grout increases tenfold from 15,000 cps to 153,000 cps in the period from 6 to 13 min after mixing. In a second experiment, the Control PPC grout was examined for its rate of change in relative viscosity. This mix also exhibited an increase in viscosity with time, having substantially greater viscosity than the PPC grout at 6 min (45,500 cps) and rising to 98,500 cps after 11 min (Fig. 8).

The effect of temperature on the relative viscosity of the PPC grout is shown in Fig. 9, in which data for the grout prepared and cured at 30°C are compared with data obtained at room temperature (~22°C). It can be seen that the relative viscosity of the grout at 30°C is approximately double that of the room temperature material at 6 min (27,500 cps and 15,000 cps, respectively) and at 9 min (90,000 cps and 51,000 cps, respectively).

These semi-quantitative measurements confirm the observations made during the field testing of the grout concerning the early thickening during pumping. They also indicate that the thickening of the grout is time and temperature dependent. There was also some indication that the laboratory prepared grout mixture had even more tendency towards early thickening than the PPC grout.

3.4.3/ Dynamic Mechanical Analysis

In order to quantify the rheological behaviour of the grout, tests were performed using a Rheometrics RDA-700 Dynamic Mechanical Analyzer (DMA) (Fig. 10). These tests were carried out to assess the time-temperature-viscosity relationships of the material, as well as observing the effect of strain rate on the behaviour of the grout. The PPC grout was analysed quantitatively at 23° and 35°C.

The DMA technique involved torsional oscillation of a layer of grout between two 40-mm diameter parallel plate aluminium disc tools, with a tool gap of 0.5 mm. One tool was stationary and attached to a torque transducer, and the other tool driven by a servomotor. The amplitude and frequency of the driven tool, as well as the temperature of the sample, were programmed and controlled by computer. The amplitude of the oscillation was expressed as (unitless) strain and the frequency (strain rate) expressed as radians per second. The viscosity was computed conventionally as the ratio of shear stress to strain rate, in units of pascal seconds (Pa.S) (see footnote previous page).

The oscillatory measurement technique has the advantage of allowing the elastic (energy storage) and the lossy (energy dissipative) subcomponents of viscosity to be separated. They are expressed as elastic modulus (G') and loss modulus (G"), both in pascals. Although the measurement was done in an oscillatory mode, the viscosity data can often be applied to pressure drop calculations in a steady-state

flow regime, such as flow in a pipe. This may be true in a limited way even for a thixotropic material such as this grout.

The PPC grout was mixed with distilled water at a w/c ratio of 0.33 using a high-shear mixer, with the grout powder added slowly to the water to avoid clumping. The time between initial mixing and the beginning of measurements was ~7 min. The first run was carried out at 23°C and 1 rad/sec, with the strain swept from 2% to 135%. The viscosity and elastic and loss moduli were plotted (Fig. 11).

It can be seen that the viscosity of the grout (η^*) dropped from 25,500 Pa.S at 2% strain to 2,000 Pa.S at ~60% strain. This twelve-fold drop in viscosity with increased strain verifies the thixotropic (pseudoplastic) nature of the grout. The final value of the viscosity was highly scattered. This may have been partially due to the sensitivity limits of the instrument, but in many of the runs the loss component was the one to scatter, and tended to scatter between two limits. This is believed to be the result of alkali attack on the aluminium tools causing some slippage during the run. (Further rheological work on the grout should be performed using stainless steel tools.)

Because of the time required for the strain sweep discussed above, the viscosity caused by the hydration of the grout was invariably combined with that caused by strain variation. Two runs were therefore conducted as time sweeps, both at 23°C and 1 rad/sec, and at strains of 1% and 20% (Fig. 12). At 1% strain, the viscosity increased from 37,000 to 300,000 Pa.S in about 10 min. Although the viscosity appeared to have reached a plateau, examination of the sample after 30 min showed it to be solidified and, presumably, slipping on the tools. At 20% strain the viscosity increased from 720 to 26,000 Pa.S in the first 6 min, and apparently dropped slightly before reaching a plateau. Again, it is suspected that the sample was slipping on the tools.

A final time sweep was attempted at 35°C, 1 rad/sec and 20% strain (Fig. 13). It can be seen that the viscosity was constant throughout the experiment at 310,000 Pa.S. Apparently, all phenomena of interest occurred in the two to three minutes prior to the start of oscillation, during which the sample temperature was coming to equilibrium (which was before the beginning of data collection).

3.4.4/ Effect of Pressure on Bleeding and Flow

The rheological tests described in the previous sections were necessarily conducted with the grout at atmospheric pressure. Since pressures during the grout injection operation in the field may reach or exceed 0.55 MPa, it was considered important to examine the effects of applied pressure on the properties of the new grout. The consequences of high pressures in a grouting system may be seen in a number of ways, including increased segregation of the grout leading to fluid loss/bleeding — an

effect which may occur through the filtration action of the steel tendon strands — and even changes in pumpability.

The relative water retentivity of the grout under pressure was determined by a modification of the American Petroleum Institute method API RP 10B used for oil-well cements [7], a method which has been recommended by Schupack [8-10] as being suitable for thixotropic grouts. The apparatus used was a 425 ml pressure filter (NL Baroid/NL Industries) fitted with a 0.3 μm glass filter and operated by applying pressure by compressed nitrogen (Fig. 14). The time to initial bleed and the water loss after 10 min at 1.03 MPa were determined (Table 2). Initial bleeding was observed immediately pressure was applied to the mixture; and 65 ml of water were collected in 10 min, although it appeared that all the available water had been expressed from the mixture by ~7.5 min. This indicates that the developmental PPC grout had relatively poor water retentivity.

The flow of the grout under applied pressure was also determined using the efflux cell arrangement shown in Fig. 15. In this method, ~200 ml of the test grout was introduced into the efflux cell. The piston was then inserted and the top plate of the cell attached before pressurizing the contents by compressed nitrogen to 0.55 MPa. At predetermined curing intervals, the efflux rate (g/s) was obtained by opening the lower valve and measuring the mass of grout delivered into a weighed container. An efflux rate of 175 g/s (avg. of two determinations) was obtained ~7-8 min after initial mixing. The significance of this value will be discussed later in relation to the properties of other formulations.

3.5/ Chemical Properties

The chemical behaviour of the PPC grout during the early hydration period was of interest to determine whether a chemical transformation (e.g., cement hydration, sulphoaluminate precipitation, alkali reaction of the aluminum, silica fume reaction, superplasticizer complexation/adsorption, etc.) was coinciding with the physical observation of thickening. Early chemical changes in the grout mixture (up to 30 min) were examined by several methods, which included: (a) heat generation, by measurement of temperature rise and time to maximum temperature; (b) rate of alkali release, as determined by pH of pore fluids obtained by high-speed centifugation of the hydrating grout mixture; (c) X-ray diffractometry (XRD), to identify which mineralogical changes were occurring in the system at early ages; and (d) thermogravimetric analysis (TGA/DTG) to estimate the degree of hydration in the system, as a function of time, and the rate of sulphoaluminate formation.

Analysis of the as-received PPC grout by XRD and TGA (Fig. 16) confirmed that the material had not deteriorated significantly during storage. Anhydrite ($CaSO_4$) with smaller quantities of gypsum ($CaSO_4 \cdot 2H_2O$) were identified as set-regulators for the Portland cement. Other minor components included ~4% calcium carbonate (permitted as a cement additive under CAN3-A5-M83), ~1.8% calcium hydroxide (presumably from some small degree of hydration during storage) and unidentified organic material (presumed to be superplasticizer). The silica fume component, being X-ray amorphous, could not be identified by these techniques.

Heat generation in the hydrating PPC grout was surprisingly slow, with a temperature rise of only +2.8°C after 68 min. By comparison, the Control PPC grout produced heat at a much greater rate, reaching +5.9°C within 14 min. As will be seen later, other formulations prepared in this study generally produced heat at rates more comparable to the Control PPC grout rather than the PPC grout.

For both the PPC and Control PPC grouts, the fluids recovered from the hydrating mixtures rapidly reached, and maintained, a pH value of 12.5–13.5 after ~5 min, as determined by pH paper. This was a common feature for all the grout mixes and indicates that the pH determination is not sensitive enough to distinguish between different formulations.

The hydration of the PPC grout was arrested at predetermined time intervals by grinding the hydrating mixture in dry acetone, followed by heating at 60°C in an atmosphere of dry nitrogen for ~1 hour [11]. This procedure closely simulates the process of 'D-drying' used by other workers and serves the dual purpose of stopping further hydration — so that a chemical 'snap-shot' of the system at different times can be obtained — and of removing physically-associated water while preserving 'chemically-bound' water (the latter being present in compounds such as calcium silicate gel, calcium hydroxide, sulphoaluminates, etc.).

XRD traces for the hydrating PPC grout pastes, quenched at various time intervals, are shown in Fig. 17. Corresponding TGA curves and calcium hydroxide and bound water contents calculated for the pastes are given in Fig. 18 and Table 3. The XRD data show that there was a very early decline in the levels of the set regulators, anhydrite and gypsum, the gypsum being almost completely consumed after 20 min hydration, and the anhydrite significantly reduced at 40 min. While the sulphate levels declined, C–S–H and calcium hydroxide, $Ca(OH)_2$, were formed by hydration of the calcium silicates (C_3S and C_2S) in the Portland cement (particularly at times after 30 mins); sulphoaluminate (AFt, ettringite) precipitation, through reaction of the calcium aluminates (C_3A and C_4AF) and sulphates, was indicated from the earliest experimental curing age (~5 min). The calculated bound water and calcium hydroxide contents of the pastes (Table 3) both showed a

corresponding marked increase between 20–40 min as hydration proceeded. These observations are all typical of events occurring in normal Portland cement concrete systems, but possibly at an increased rate. It should be noted that, in the present system, the calcium hydroxide levels will also be affected (i.e., reduced) by its participation in the pozzolanic reaction with the silica fume.

3.6/ Summary of Properties

Physically, in comparison with the materials reported in the Queen's study [3,4], the prepackaged PPC grout had similar expansion, slightly lower strength but a considerably longer flow time (Table 2). The PPC grout also had poor water retentivity. The differences in strength and flow are considered to be more related to the methods of mixing (i.e., low-shear vs. high-shear), rather than gross differences in the materials themselves. However, it should be kept in mind that the Queen's grout was batched in the laboratory, whereas the PPC grout was of commercial origin.

The PPC grout was rheologically complex, with both thixotropic (pseudoplastic) behaviour at room temperature, and a time-dependent tendency to "lock up" very quickly if not kept flowing. Similar behaviour was observed in the PPC Control. If allowed to remain locked up for a few minutes, the grout solidified to the point where flow could not be easily induced. This solidification was seen in the fast initial rise in viscosity observed in the instrumental results. In contrast, if the grout was stirred steadily, it failed to solidify: a sample stirred for 4 hours at room temperature showed only a minor increase in viscosity. However, small amounts of material removed from the stirred mixture at intervals throughout the 4-hour time period rapidly solidified after a few minutes if undisturbed.

The rheological behaviour of the PPC grout is considered to be largely of chemical origin and to be consistent with the formation of hydration products which rapidly interlock to form a network. This structure would be initially very weak and easily broken down by shear forces, only to reform when the disturbance was removed — and thus manifested as thixotropy. Only a small amount of reaction appeared necessary to form a perceptible binding network.

Two points relating to chemical changes in the system are of possible significance to the formation of the network. First, there was a strong indication that the thickening behaviour was temperature dependent — the initial weak network (thixotropy) apparently formed almost immediately at room temperature, but took more than an hour to form at 0°C; whereas at 35°C the grout formed a very stiff, irreversible structure within 3 min. Second, the PPC grout was slow to release heat, suggesting that the process responsible for the thixotropy may not be as strongly exothermic as cement hydration.

While the data at this stage are incomplete, four of the factors noted earlier (page 5) are possible candidates for the thickening phenomenon:

- formation of sulphoaluminate (AFt, ettringite)
- reactivity of the silica fume
- incompatibility of the superplasticizer
- gas generation by the aluminum powder

There was evidence for the rapid formation of ettringite by the grout; however, it was not excessive, and since ettringite is precipitated in the form of thin crystals, it would not be expected to reform after shearing (i.e., it is not reversible); therefore, it is unlikely to be related to the thixotropic behaviour, although it would contribute to time-dependent thickening.

A more likely candidate in this regard is the silica fume which can react with water to form a gel:

$$ySiO_2 + 3H_2O \rightarrow ySiO_2 \cdot 3H_2O \overset{\text{shear}}{\leftrightarrow} ySiO_2 \cdot 3H_2O$$

(silica) (water) (silica gel) (silica sol)

This reaction may occur very rapidly by virtue of the high surface area of the silica fume and the highly alkaline environment (pH 13.5) produced by the release of alkalis from the hydrating cement. Silica gel formed in this way would also be very reactive and capable of reacting further with cement alkalis and lime to form calcium silicate hydrates (C–S–H):

$$ySiO_2 \cdot 3H_2O + \chi Ca(OH)_2 \rightarrow \chi CaO \cdot ySiO_2 \cdot (\chi + 3)\, H_2O$$

(C–S–H)

A reacting system of this type could produce both thixotropy and thickening: the gel would be responsive to shear and capable of undergoing reversible transformations from the viscous "gel" form to the lower viscosity "sol" form; and the developing C–S–H would produce structure and solidification. From the TGA data, it appears that the gel is formed almost immediately the silica fume comes into contact with water; the C–S–H, as indicated by the sudden rise in bound water, seems to commence formation at 20–30 min.

The contribution that superplasticizer incompatibility may make to the early thickening is not clear from the initial chemical data, although as will be evident in the next section, it may be the one of the main problems with the PPC grout. The thickening does not seem to be related to air entrainment or gas generation.

4/ Screening Tests to Modify Grout Formulation

4.1/ Design Criteria

The second stage of the investigation was concerned with modification of the grout formulation for improved rheological performance and involved the following activities: (a) examination of the effects of changes in the mix components (Portland cement, superplasticizer, silica fume, aluminum powder); (b) examination of the effects of temperature and pressure on the flow properties of the grout; and (c) compliance testing of an optimized formulation. The target specifications for post-tensioning grout materials are summarized in Table 1.

An extensive array of tests were conducted on the various modified and reference grout formulations to monitor the changes in both the setting and rheological properties with changes in the grout composition. These tests serve as a common reference for comparison of the various experimental formulations with the PPC material, and are as follows:

- **Grout setting properties:**
  - –expansion
  - –bleeding
  - –time to initial set
  - –maximum temperature rise
  - –time to maximum temperature
  - –solution pH

- **Grout flow and rheological properties:**
  - –slump
  - –flow cone (non-standard)
  - –consistency
  - –viscosity change with time (Brookfield RVT Viscometer)
  - –efflux rate at 0.55 MPa pressure

The main goal in the above testing procedures was to accumulate sufficient data on a large number of mix designs in order to isolate the main factors contributing to the observed thickening of the PPC packaged grout material. The results of these screening tests allow for selection of an optimum grout formulation based on knowledge of the interdependency of the four component materials.

4.2/ Materials

The materials used for the screening tests were all available commercially in Ontario, and are briefly described below.

The Portland cements that were used in the optimization trials — four CSA Type 10 cements and one Type 50 (low Al_2O_3) cement — were from four different Ontario manufacturers. The chemical, physical and strength characteristics of the five cements are given in Table 4. The Portland cements used in this study were certified free of false set.

The silica fume used in this program was from a source in Bécancour, Québec, the same as that used in the PPC packaged material. No attempt was made in this study to examine the effects of different sources of silica fume, as it is believed that silica fume from different manufacturers will behave in essentially the same manner in this type of grout fomulation.

Three sources of aluminum powder (with particle size distributions containing approximately 15% +325 mesh material), both coated and uncoated, were selected from commercial suppliers:

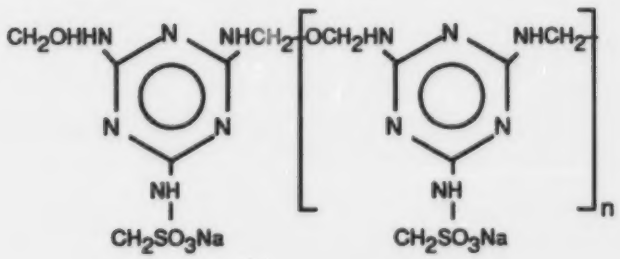
(a) Source 1, uncoated pharmaceutical grade
(b) Source 2, uncoated
(c) Source 3, coated

The Source 1 aluminum powder corresponds to the material used in the PPC packaged material, but was not available for use in this program. For this reason, the Source 2 (uncoated) and Source 3 (coated) materials were used for all laboratory prepared trial formulations. As mentioned earlier, the coated aluminum is used in order to slow the release of expansive gases in the hydrating grout, and was used to gauge the effect of early versus later gas evolution.

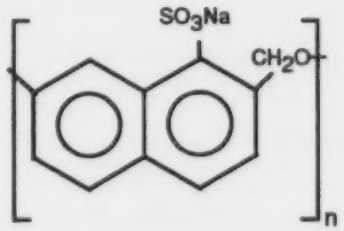
The five superplasticizers used in the grout formulations were obtained from commercial sources, and are designated as follows:

Type of Superplasticizer	Designation
Control (SMF type)	SP1
Naphthalene sulphonate (SNF type) (retarded, 2% Na-gluconate)	SP2
Naphthalene sulphonate (non-retarded)	SP3
Melamine-formaldehyde + naphthalene sulphonate (80:20)	SP4
Melamine-formaldehyde + Na-lignosulphonate (90:10)	SP5

The structures of the control SMF (sulphonated melamine formaldehyde-based) and the SNF (sulphonated naphthalene formaldehyde-based) superplasticizers are shown as follows:



Sodium salt of sulphonated melamine formaldehyde



Sodium salt of sulphonated naphthalene formaldehyde

4.3 / Optimization Trials

The formulations used for the initial screening tests are given in Table 5. In addition to the reference PPC and PPC Control materials, a series of formulations incorporating various types of Portland cement, superplasticizer, aluminum powder and amounts of silica fume was examined in order to isolate the effects of the various components with respect to the physical characteristics of the grout. The rheological properties of a further series of mixes were examined semi-quantitatively using the Brookfield RVT Viscometer (see Section 3.4.2). The compositions of these mixes are presented in Table 6. The following sections will outline the effect of the various materials on the properties of the grout, and from this data an optimum formulation will be selected.

4.3.1/ Effect of Cement

The effect of cement type (all formulations compared contained the SP1 superplasticizer) on the setting and rheological properties of the grout are shown in Tables 7 and 8. The A10 cement (which was also used in the PPC Control sample) showed the lowest expansion and lowest slump values among the cements tested, as well as the lowest flow times (along with the PPC material) in the flow cone tests. These data suggest that the A10 cement, at least in combination with the SP1 superplasticizer, has an inherent tendency toward decreased flow as compared to the other cements when used in this type of mixture. In contrast, A50, the Type 50 cement from the same manufacturer, seemed to exhibit good flow properties under these conditions. Of note was the

significantly lower pressurized efflux rate measured for the PPC packaged material as compared to the other mixtures, suggesting that this formulation may prove more difficult to pump.

The relative viscosities (with respect to time) of mixtures containing the four Type 10 cements are compared to the PPC and PPC Control mixtures in Fig. 19. As can be seen, the data consists of two distinct groups: the first, being the PPC and PPC Control (both containing the SP1 superplasticizer); and the second, being the mixes containing the A10, B10, C10 and D10 cements in combination with the SP3 superplasticizer. The A10 mixture, however, did exhibit a noticeably higher viscosity at all test times than do the B10, C10 and D10 mixtures, although not as high a value as is measured for the PPC materials. The B10, C10 and D10 containing mixes all exhibited a lower initial viscosity and a lower viscosity increase over the testing period than do the mixes containing the A10 cement (which includes the PPC materials). The effect of the superplasticizer, which is already apparent from the above data, will be discussed in more detail in Section 4.3.3.

The data presented above suggest that the type of Portland cement used in the grout formulation is an important factor with regard to potential thickening effects. It appears that the Type 10 cement from manufacturer "A" is particularly affected in this regard.

4.3.2/ Effect of Silica Fume and Aluminum Powder

Silica fume is added to the grout in order to increase strength and lower porosity, both of which are desirable for this type of material. However, the silica fume increases water demand, necessitating the use of a superplasticizer to obtain the desired water to solids ratio. A limited number of mixes were examined to assess the effect of the silica fume on the grout (Tables 9 and 10). The silica fume was seen to reduce the setting time and the slump of the mixture, although there appeared to be little effect on the efflux under pressure. This suggested that the silica fume was itself not a major factor in the early thickening observed in the PPC material.

The effects of the aluminum powder on the properties of the grout are presented in Tables 11 and 12. The only property which is significantly affected by the absence of aluminum powder was the measured expansion of the grout, which became a shrinkage of 3% in the absence of aluminum powder. Figure 20 shows that marginally lower relative viscosity values were obtained in the absence of aluminum powder, but when compared to the curves for the PPC materials it is apparent that this difference is relatively minor. As with the silica fume, the source and type (i.e., coated or uncoated) of the aluminum powder is not believed to be a significant factor in the thickening of the PPC grout.

4.3.3/ Effect of Superplasticizer

The effects of the various superplasticizers (at addition levels of 1%) on the setting and rheological properties of grout mixes using the A10 cement are shown in Tables 13 and 14. Of the five superplasticizer compositions examined, the SP2 and SP3 materials are clearly superior in terms of slump and flow cone results in systems using the A10 cement. SP5 is clearly unacceptable, as no flow in the modified flow cone test was recorded, and SP1 and SP4 were significantly inferior to SP2 and SP3 in this test. Of note is the increased setting time required for the SP2 material (a retarded sulphonated naphthalene formaldehyde), indicating that the retarding admixture (sodium gluconate) has some effect in this system.

In Fig. 21 a comparison is made in the relative viscosities of mixtures using 1% SP1, 4% SP1 and 1% SP3 in mixtures containing three cement types (A10, A50 and B10). With the A10 cement, the SP3 containing mixture shows a significantly lower viscosity than the mixes containing 4% SP1, which is itself superior to the mix containing only 1% SP1. The trend is repeated for the A50 cement, although at slightly reduced viscosity levels. With the B10 cement, a noticeable drop in the viscosity of the grout is observed when the SP1 level is increased from 1% to 4%, and the values using SP3 are essentially the same as those obtained for 4% SP1.

Three significant conclusions can be reached from the data discussed above:

- The relative viscosities of grout mixes containing the A10 Portland cement fall appreciably when the SP3 superplasticizer is used in place of the SP1 superplasticizer.
- The relative viscosities of grouts containing the B10 cement are much lower than similar mixes containing the A10 cement and the same superplasticizer.
- The SP3 superplasticizer produces noticeably lower relative viscosities than the SP1 plasticizer when used at the same addition level, regardless of cement type.

At this point, it was apparent that a formulation containing the B10 cement, in combination with the SP3 superplasticizer, was a likely candidate as the optimum grout formulation. For this reason, the "B10-SP3" formula was selected for evaluation against the PPC and PPC Control grouts with regard to its rheological behaviour at elevated temperatures and pressures, as a final step in selecting an optimum formulation.

4.3.4/ Temperature and Pressure Effects

As was discussed in Section 2, the on-site temperature regime for grouting operations can regularly raise grout temperatures to 30°C or higher, so testing of any potential formulation at elevated temperatures is deemed essential. Figure 22 shows the relative viscosity measurements over time for the PPC grout compared to the B10-SP3 mixture at 23°C and 30°C. The temperature had virtually no effect on the B10-SP3 mixture, while the viscosity of the PPC material increased at a greater rate at 30°C as compared to 23°C. Similarly, the efflux rate of the A10-SP3 grout at 0.55 MPa was higher than that of the PPC material at both 25 and 30°C (Table 15). At elevated temperature and pressures, the B10-SP3 formulation clearly outperformed the PPC prepackaged material.

4.3.5/ Selection of Optimum Formulation

The screening program, described above, leads to the following general conclusions with regard to the developmental PPC grout formulation:

- The cement and superplasticizer used in the PPC grout formulation appear to be incompatible when incorporated in a cement/silica fume/aluminum powder/ superplasticizer combination.
- The SMF-type (SP1) superplasticizer was found to be less effective in terms of the measured flow and rheological properties in the grout formulations examined than were the SNF-type (SP2 or SP3) superplasticizers.
- The B10 cement, in combination with the SNF-type (SP3) superplasticizer, proved to exhibit satisfactory setting and rheological properties, and also performed well in terms of relative viscosity and pressurized flow characteristics, as compared to the PPC materials, at both ambient and elevated temperatures.

The selected optimum formulation consists of the B10 Portland cement and the SP3 superplasticizer along with silica fume and aluminum powder in the same proportions as was specified for the PPC packaged material. The selection of the B10 cement was somewhat arbitrary in that either the C10 or D10 cements would appear to work equally well in the grout system. The important factor that must be considered in this or any formulation is that the source of the Portland cement can have a significant effect on the properties of the final product. In order to avoid the problems encountered with the PPC packaged material, all of the grout components which will be used for full-scale operations must be tested under simulated field conditions (i.e., temperature, mixer shear, time factors, etc.). In the following section, the testing of the optimized grout formulation in accordance with the target specifications (Table 1) will be discussed.

5/ Testing of Modified Formulation

A comparison of the properties of the PPC grout with those of the optimized grout formulation (B10-SP3) is given in Table 16, together with the target specifications where applicable.

5.1/ Physical Properties

The strength development of the PPC grout and the optimized grout formulation up to age 28 days are given in Fig. 23. The optimized grout showed superior strength at 7 and 28 days as compared to the PPC grout, although it should be noted that strength developments of both formulations exceed the target specifications by a considerable margin. The values obtained for expansion for both the PPC and optimized formulation were within the target range for this type of material.

The results for water retentivity at 1.03 MPa showed a marked difference between the PPC grout and the optimized grout, although neither material exhibited any tendency towards static (i.e., unpressurized) bleeding. The PPC material released 24% of its available water under this pressure, whereas the optimized grout released only 14% of its available water. This fact suggests that the PPC grout is not binding water to the same extent as the optimum grout at early ages, a result that was supported by the low measured temperature increase for the PPC material (2.8°C) as compared to the optimum grout (6.2°C). The lack of temperature increase in the PPC material is interpreted as a lack of cement hydration reactions occurring in the grout, which can result in more free water being available. The availability of free water in the grout can, in turn, lead to segregation of the solid and fluid portions of the grout under the influence of high pressures.

These factors indicate that aside from the thickening problems encountered by the PPC material, there is a distinct possibility of it segregating under pressure in the tendon ducts. Whether this actually occurs under field conditions and pressures has not been determined, but it is clear that the optimized grout formula will be less susceptible to this effect.

5.2/ Rheological Properties

The flow time, efflux rate, consistency and slump values for the optimized grout were all improved compared to those of the PPC material. The flow time (ASTM C 939) of the optimized grout was much lower than that of the PPC material, and fell within the target range. That the flow time of the PPC material was so high is indicative of its thixotropic behaviour, as this is a static test which allows

sufficient time for the thickening effect to manifest itself. The short flow time of the optimized grout indicates that there is no problem with this formulation in terms of thickening behaviour.

The efflux rate at 0.55 MPa of the optimized grout was considerably better than that obtained for the PPC material, and this superiority was also demonstrated at elevated temperatures (see Table 15).

5.3/ Durability Related Properties

The electrical resistivity, chloride permeability and corrosion susceptibility of the optimized grout, as compared to the PPC material, were found as follows:

Property	Target	PPC Grout	Optimized Grout
Chloride Permeability (Coulombs)	100–1,000	2,677	2,740
Electrical Resistivity (ohm-cm):	>10,000		
3 days	-	4,250	3,180
7 days	-	9,920	7,050
14 days	-	24,100	16,300
21 days	-	112,000	59,700
28 days	-	104,000	116,000
200 days*	-	64,100	57,300
Corrosion Susceptibility:			
Potential Difference (V)	-	-0.086	-0.083
Current Flow ($\mu A/cm^2$)	<1	1.1	0.6

The chloride permeability of both the PPC grout and the optimized grout appeared higher than desired. By comparison, the value quoted in Hope and Ip's report for the 10% silica fume grout was 702 coulombs [3]. The reason for this difference is not clear but may again be related to the method used for mixing the grout for testing (see Section 3.3). The high-shear mixing used in the present study may produce a more uniform, interconnected pore structure, which could account for the increased chloride permeability.

The electrical resistivities of both materials exceeded the target value after 14 days, and continued to do so for the entire length of the test (Fig. 24). After being immersed in a 3.5% NaCl solution for 200

* 200 days in 3.5% NaCl solution

days, the electrical resistivity values for both materials fell considerably, although both still well exceeded the target value of 10,000 ohm-cm.

The corrosion susceptibility test results showed that the optimized grout met the target current flow (less than 1 $\mu A/cm^2$), while the current flow of the PPC grout was slightly above this value. Again, the test results were slightly higher in this study than those obtained by Hope and Ip [3,4], suggesting that the mixing action has an effect on these durability-related parameters.

6/ Conclusions and Recommendations

The thickening of the PPC packaged grout material has been examined by several qualitative and quantitative techniques, and its early rheological behaviour was found to closely resemble that of a classical thixotropic (pseudoplastic) fluid. That similar behaviour was observed with a laboratory blend prepared with the same raw materials as the PPC packaged material, served to confirm that the thickening was an inherent property of the formulation, and not due to possible errors in batching of the PPC packaged material.

Physical, chemical, and mineralogical probes indicated that the thixotropy of the grout was probably associated with hydration of the silica fume component. In addition, a non-reversible thickening was also observed, consistent with hydration and reaction of silica gel with cement, as well as some contribution from sulphoaluminate (AFt) precipitation. The thickening was considered to be of chemical origin, rather than a physical effect common to most thixotropic systems. The outcome is that although the thickening may be reversible at very early ages, re-fluidizing the system at later ages proves increasingly difficult as the hydration reactions proceed and a chemically bonded silicate network is formed.

From data accumulated during the site visit, a set of experimental parameters was established which defined the important factors affecting the rheological properties of the grouting material. The most significant factors were found to be the shear rate of the mixer used in the preparation of the grout and the temperature and pressure at which the grout is handled. For this reason, a carefully controlled mixing regime was established, as well as careful monitoring of the ambient and grout temperatures, during testing.

A series of tests was conducted in which the four components — i.e., cement, silica fume, superplasticizer and aluminum powder — were varied, using the same w/c ratio (0.33) in each case. The following conclusions were reached from these tests:

- The grout was very sensitive to the type of superplasticizer used. The SMF-type (SP1) superplasticizer, as used in the PPC material, was less effective than either of the SNF-type materials (SP2 or SP3), and was particularly ineffective when used in combination with the A10 cement.

- The Portland cement type used was found to be a significant factor, in that formulations incorporating the A10 cement (as used in the PPC grout) exhibited reduced flow compared to similar mixes using alternative cements. The flow properties of the grout are extremely sensitive to the origin of the cement used.

- The silica fume affected the grout, in that its absence aided in flowability, although the resultant increase in both porosity and permeability would prove unacceptable. Silica fume hydration is believed to contribute to the thixotropy and thickening mechanism (see above).

- The type of aluminum powder used (coated vs. uncoated) had little affect on the rheological properties of the grout. Its presence, however, was necessary to achieve the desired expansion characteristics.

It has been concluded that the main problem with the PPC packaged grout was an incompatibility of the particular combination of Portland cement and superplasticizer used in the formulation. At this stage, however, the underlying chemical cause of this incompatibility remains unclear.

An optimized grout mixture consisting of the source "B" Type 10 Portland cement and the SP3 (i.e., non-retarded naphthalene sulphonate) superplasticizer was selected, and subsequently tested for compliance with the design criteria. The optimized grout exhibited superior flow properties to those of the PPC reference grout, at both ambient and elevated temperatures and in both static and pressurized configurations. Improved strength results were also obtained for the optimized grout. The chloride permeability and electrical resistivities of the two grout materials were quite comparable. However, the optimized grout appeared to have reduced corrosion susceptibility compared to the PPC material (0.6 and 1.1 $\mu A/cm^2$, respectively). Comparison of these data with those obtained by Hope and Ip [3,4] suggests that there are differences in the measured values of these durability parameters, possibly related to the method (i.e., shear) used for mixing.

The water retentivity of the optimized grout was improved compared to the PPC material, implying that it will be less susceptible to segregation while being injected into tendon ducts.

The following general recommendations are made with respect to continued development and testing of the optimized cement grout formulation:

- That the viscosity/shear dependence of the optimized grout be further investigated with large sample sizes using an adaptation of the two-point method used for concrete. Examination of larger samples will be important in identifying potential mass-related effects (e.g., heat of hydration).

- That the optimized grout be tested under simulated field conditions using a pilot-scale (e.g., 200-metre) pumping loop designed to measure pressure drop, flow rate and temperature.

- That the mechanism of the cement/superplasticizer sensitivity be investigated.

- That specifications be developed for the Portland cement to be used in the grout, with due regard for its sensitivity to the superplasticizer and other components.

References

[1] Taylor, R.W., "Grouting of Ducts in Post-Tensioned Prestressed Concrete," *TRRL Contractor Report 24* (Crowthorne, Transport and Road Research Laboratory, Dept. of Transport, U.K., 1986), 24 pp.

[2] Woodward, R.J., "The Grouting of Five Prestressed Concrete Bridges During Construction," *TRRL Supplementary Report 668* (Crowthorne, Transport and Road Research Laboratory, Dept. of the Environment/Dept. of Transport, U.K., 1981), 20pp.

[3] Hope, B.B., and Ip, A.K.C., "Improvements in Grouts and Grouting Techniques for Cable Ducts," *Technical Report No. ME-87-04*, Research and Development Branch, Ontario Ministry of Transportation and Communications, Downsview, March 1987, 40 pp.

[4] Hope, B.B., and Ip, A.K.C., "Grout for Post-Tensioning Ducts," *ACI Materials J.*, July-Aug. 1988, pp. 234-240.

[5] Information provided by H. Schell, Ontario Ministry of Transportation, 1988.

[6] Information provided by T. Cantlon, Ontario Ministry of Transportation, 1988.

[7] API RP 10B: "API Recommended Practice for Testing Oil-Well Cements and Cement Additives" (American Petroleum Institute, TX, Dec. 1979).

[8] Schupack, M., "Water-Retentive Admixture for Grouts Serve Post-Tensioning Needs," *Concrete Construction*, Jan. 1984, pp. 47-51.

[9] Schupack, M., "Admixture for Controlling Bleed in Cement Grout Used in Post-Tensioning," *PCI Journal*, Nov.-Dec. 1974, pp. 28-39.

[10] Schupack, M., "Grouting Tests on Large Post-Tensioning Tendons for Secondary Nuclear Containment Structures," *PCI Journal*, March-April 1971, pp. 85-97.

[11] Hemmings, R.T., and Douglas, E., "Characterization of Granulated Blast Furnace Slag for Portland Cement Replacement," *Proc. Int. Workshop on Granulated Blast-Furnace Slag in Concrete, Toronto*, Oct. 22-23, 1987, pp. 439-458.

Table 1/ Target specifications for post-tensioning grout

Property	Target
Fineness	>95%, -45 μm
Flow time (ASTM C 939)*	12–20 seconds
Pumpability*	Pumpable at 0.55 MPa
Bleeding (15 min stand) (ASTM C 940)	None
Segregation (0.55 - 1.03 MPa)*	None
Expansion (plastic) (ASTM C 940)	8±2% (at initial set)
w/c	<0.40
Strength (50 mm cubes, restrained):	
7 – day	>20 MPa
28 – day	>35 MPa
Set time	~2–4 hours
Chloride content	≤0.025%
Chloride permeability (AASHTO T227-831)	100–1000 coulombs
Electrical resistivity	>10,000 ohm-cm
Corrosion susceptibility	<1 μA/cm^2

* *See text*

Table 2/ Comparison of physical and chemical properties of reference grouts

Test	PPC Reference Grout [this study]	Hope and Ip [refs. 3 & 4]
Flow time (ASTM C 939), sec	55	13.9
Efflux time, at 0.55 MPa, g/sec	175	..
Consistency, penetration, mm	34	..
Slump, patty diam, mm	233	..
Expansion (ASTM C 940), %	9	7.0
Bleeding (ASTM C 940)	none	none
Water retentivity:		
Time to initial bleed, sec	0	..
Water loss, 10 min at 1.03 MPa, ml	65*	..
Initial set, hr:min	4:52	..
Compressive Strength, MPa		
1/day	28.1	..
3/day	34.0	..
7/day	39.8	48.9
14/day	48.4	..
28/day	47.8	62.1
Time to max. temperature, hr:min	1:08	..
Temperature rise, °C	2.8	..
pH	12.5-13.5	12.5-13.5

* *no further loss of water observed after 7.5 min.*

Table 3/ Bound water and calcium hydroxide contents of hydrating PPC grout*

Time of Hydration (min)	Bound Water (%)	Calcium Hydroxide (%)
7.5	2.3	1.9
10	2.6	2.0
15	2.6	2.1
20	2.6	1.5
25	3.2	1.9
30	3.1	1.9
40	6.6	2.7
60	4.8	2.9

* *Calculated from thermal traces (TGA/DTG)*

Table 4/ Chemical and physical properties of portland cements*

	Cement Source and Type†				
	A-10	A-50	B-10	C-10	D-10
Chemical Composition:					
SiO_2	19.5	21.3	20.3	22.1	20.7
Al_2O_3	4.1	3.7	5.7	4.2	5.8
Fe_2O_3	3.1	4.3	2.2	2.9	2.4
CaO	61.0	61.9	62.6	62.6	61.8
MgO	4.2	4.3	2.4	3.3	2.3
Na_2O	0.52	0.45	..	..	0.23
K_2O	0.90	0.85	..	..	1.11
SO_3	3.99	2.39	3.00	3.00	4.31
Total alkalies (as Na_2O)	1.11	1.01	0.93	0.42	0.96
Insoluble residue	..	..	0.46	0.20	..
Free lime	..	..	0.78	0.51	0.85
Loss on ignition	2.38	0.85	2.45	1.40	0.81
Bogue Composition:					
C_3S	56.8	52.5	50.3	45.9	39.8
C_2S	13.0	21.5	20.4	28.7	29.4
C_3A	5.7	2.6	11.4	6.3	11.1
C_4AF	9.4	12.9	6.6	8.8	7.4
C_3S/C_3A	10.0	20.2	4.4	7.3	3.6
Physical Properties:					
Fineness, % +45 µm	..	..	17.7	10.2	..
Blaine (m^2/kg)	..	..	363	398	368
Initial set (min)	..	..	125	127	113
Air content (%)	..	..	9.6	9.1	..
Expansion (%)	..	..	0.13	0.09	..
Compressive Strength (MPa):					
1 day	..	..	13.8	..	..
3 day	..	..	24.4	23.5	23.2
7 day	..	..	29.6	29.6	30.7
28 day	..	..	35.5	40.5	41.7

* *Source: manufacturers' data;*

† **A, B, C** *and* **D** *denote commercial Portland cement sources;*
10 *= Normal Portland cement (Type 10);* **50** *= Sulphate-Resistant Portland cement (Type 50).*

Table 5/ Mix formulations for grout screening tests

Mix Designation	Mix Composition			
	Cement	Silica Fume	Superplasticizer	Aluminum Powder
PPC reference	A10	10%	SP1	Source 1
PPC control	A10	10%	SP1	Source 2
A10-SP1	A10	10%	SP1	Source 2
A10-SP1(0)	A10	none	SP1	Source 2
A10-SP1(8)	A10	8%	SP1	Source 2
A10-SP1	A10	10%	SP1	Source 3
A50-SP1	A50	10%	SP1	Source 2
A10-SP1(N)	A10	10%	SP1	None
A10-SP2	A10	10%	SP2	Source 2
A10-SP3	A10	10%	SP3	Source 2
A10-SP4	A10	10%	SP4	Source 2
A10-SP5	A10	10%	SP5	Source 2
B10-SP1	B10	10%	SP1	Source 2
C10-SP1	C10	10%	SP1	Source 2
D10-SP1	D10	10%	SP1	Source 2

Sample Coding: **X-##-SP#(Y)**

X## = *Cement Type (See Table 4)*
SP# = *Superplasticizer Type (See Section 4.2)*
(Y) = *Denotes either no aluminum powder in sample (N) or % silica fume when not 10% (#%)*

Table 6/ Mix formulations for rheological screening tests

Mix Designation	Mix Composition			
	Cement	Silica Fume	Superplasticizer	Aluminum Powder
PPC reference	A10	10%	SP1	Source 1
PPC control	A10	10%	SP1	Source 3
A50-SP3	A50	10%	SP3	Source 2
A10-SP3	A10	10%	SP3	Source 2
A50-SP3	A50	10%	SP3	Source 3
A10-SP3	A10	10%	SP3	Source 3
A10-SP3	A10	10%	SP3	Source 3
A10-SP3	A10	10%	SP3	None
A50-SP3	A50	10%	SP3	None
C10-SP3	C10	10%	SP3	Source 3
D10-SP3	D10	10%	SP3	Source 3
B10-SP3	B10	10%	SP3	Source 3
A10-SP1 (4%)	A10	10%	4% SP1	Source 3
B10-SP1	B10	10%	SP1	Source 3
B10-SP1 (4%)	B10	10%	4% SP1	Source 3
A50-SP1	A50	10%	SP1	Source 3
A50-SP1 (4%)	A50	10%	4% SP1	Source 3

* *Sample Coding:* **X##-SP#**

X## = *Cement Type (See Table 4)*
SP# = *Superplasticizer Type (See Section 4.2)*
4% = *Sample containing 4% superplasticizer*

Table 7/ Effect of cement type on the setting properties of grout trial mixes (SMF Superplasticizer)

Mix	Expansion (%)	Bleeding	Initial Set (hr:min)	Max Temp. Time (hr:min)	Temp Rise (°C)	pH
PPC reference	5.5	Nil	4:52	1:11	2.8	13.5
PPC control	2	Nil	4:20	0:50	6.6	13.5
A10-SP1	5	Nil	4:17	0:14	5.9	13.5
A50-SP1	14	Nil	5:15	0:31	3.7	13.5
B10-SP1	14	Nil	4:45	0:33	4.5	13.5
C10-SP1	12	Nil	5:21	0:41	4.0	13.5
D10-SP1	..	Nil	5:29	0:33	4.5	13.5

Table 8/ Effect of cement type on the rheological properties of grout trial mixes (SMF Superplasticizer)

Mix	Slump (patty dia. mm)	Flow Cone (g/s)	Consistency (mm penetration)	Efflux Rate (g/sec)
PPC reference	219	10.0	33	175
PPC control	150	10.0	29	272
A10-SP1	155	14.4	29	299
A50-SP1	245	24.5	28	286
B10-SP1	215	22.6	35	308
C10-SP1	300	35.7	41	325
D10-SP1	310	30.8	45	327

Table 9/ Effect of silica fume on the setting properties of grout trial mixes (Cement A10)

Mix	Expansion (%)	Bleeding	Initial Set (hr:min)	Max Temp. Time (hr:min)	Temp Rise (°C)	pH
PPC reference	5.5	Nil	4:52	1:11	2.8	13.5
PPC control	2	Nil	4:20	0:50	6.6	13.5
A10-SP1 (0)	11	Nil	>5:10	0:30	5.0	13.5
A10-SP1 (8)	12	Nil	4:25	0:28	6.4	13.5
A10-SP1 (10)	5	Nil	4:17	0:14	5.9	13.5

( ) denotes silica fume %

Table 10/ Effect of silica fume on the rheological properties of grout trial mixes (Cement A10)

Mix	Slump (patty dia. mm)	Flow Cone (g/s)	Consistency (mm penetration)	Efflux Rate (g/sec)
PPC reference	219	10.0	33	175
PPC control	150	10.0	29	272
A10-SP1(0)	260	20.1	50	294
A10-SP1(8)	165	21.0	31	291
A10-SP1(10)	155	14.4	29	299

( ) denotes silica fume %

Table 11/ Effect of aluminum powder on the setting properties of grout mixes (Cement A10/Smf Superplasticizer)

Mix	Expansion (%)	Bleeding	Initial Set (hr:min)	Max Temp. Time (hr:min)	Temp Rise (°C)	pH
PPC reference	5.5	Nil	4:52	1:11	2.8	13.5
PPC control	2	Nil	4:20	0:50	6.6	13.5
A10-SP1 (None)	-3	Nil	4:18	0:39	3.9	13.5
A10-SP1 (uncoated)	5	Nil	4:17	0:14	5.9	13.5
A10-SP1(coated)	13	Nil	4:20	0:34	5.9	13.5

( ) denotes type of aluminum powder

Table 12/ Effect of aluminum powder type on the rheological properties of grout trial mixes (Cement A10/SMF Superplasticizer)

Mix	Slump (patty dia. mm)	Flow Cone (g/s)	Consistency (mm penetration)	Efflux Rate (g/sec)
PPC reference	219	10.0	33	175
PPC control	150	10.0	29	272
A10-SP1 (None)	155	18.6	32	292
A10-SP1 (uncoated)	155	14.4	29	299
A10-SP1 (coated)	160	22.5	24	288

( ) denotes type of aluminum powder

Table 13/ Effect of superplasticizer type on the setting properties of grout trial mixes (Cement A10)

Mix	Expansion (%)	Bleeding	Initial Set (hr:min)	Max Temp. Time (hr:min)	Temp Rise (°C)	pH
PPC reference	5.5	Nil	4:52	1:11	2.8	13.5
PPC control	2	Nil	4:20	0:50	6.6	13.5
A10-SP1	5	Nil	4:17	0:14	5.9	13.5
A10-SP2	9	Nil	4:56	0:47	5.0	13.5
A10-SP3	9	Nil	4:35	0:35	5.4	13.5
A10-SP4	10	Nil	3:49	1:10	2.3	13.5
A10-SP5	9	Nil	4:20	0:24	6.3	13.5

Table 14/ Effect of superplasticizer type on the rheological properties of grout trial mixes (Cement A10)

Mix	Slump (patty dia. mm)	Flow Cone (g/s)	Consistency (mm penetration)	Efflux Rate (g/sec)
PPC reference	219	10.0	33	175
PPC control	150	10.0	29	272
A10-SP1	155	14.4	29	299
A10-SP2	235	27.3	38	263
A10-SP3	227	22.5	32	307
A10-SP4	155	15.9	33	313
A10-SP5	107	no flow	30	..

Table 15/ Effect of temperature on the efflux rate of the PPC and optimized (B10-SP3) grout mixtures

Mixture	Efflux Rate at 25°C (g/sec)	Efflux Rate at 30°C (g/sec)
PPC reference grout	203	237
Optimized grout (B10-SP3)	323	270

Table 16/ Summary of physical and chemical properties of optimized grout

Test	PPC Ref. Grout	Optimized Grout	Target
Flow time (ASTM C 939), sec	55	15.2	12-20
Efflux rate, at 0.55 MPa, 25°C, g/sec	175	323	..
Consistency, penetration, mm	34	43	..
Slump, patty diam, mm	233	305	..
Expansion (ASTM C 940), %	9	10	8±2
Bleeding (ASTM C 940)	none	none	none
Water retentivity:			
Time to initial bleed, sec	0	~5	..
Water loss, 10 min at 1.03 MPa, ml	65*	36.5	..
Initial set, hr:min	4:52	4:44	..
Compressive strength, MPa			
1/day	28.1	23.8	..
3/day	34.0	35.7	..
7/day	39.8	48.1	>20
14/day	48.4	49.6	..
28/day	47.8	51.6	>35
Time to max. temperature, hr:min	1:08	0:59	..
Temperature rise, °C	2.8	6.2	..
pH	12.5-13.5	12.5-13.5	..

* *no further loss of water observed after 7.5 min.*

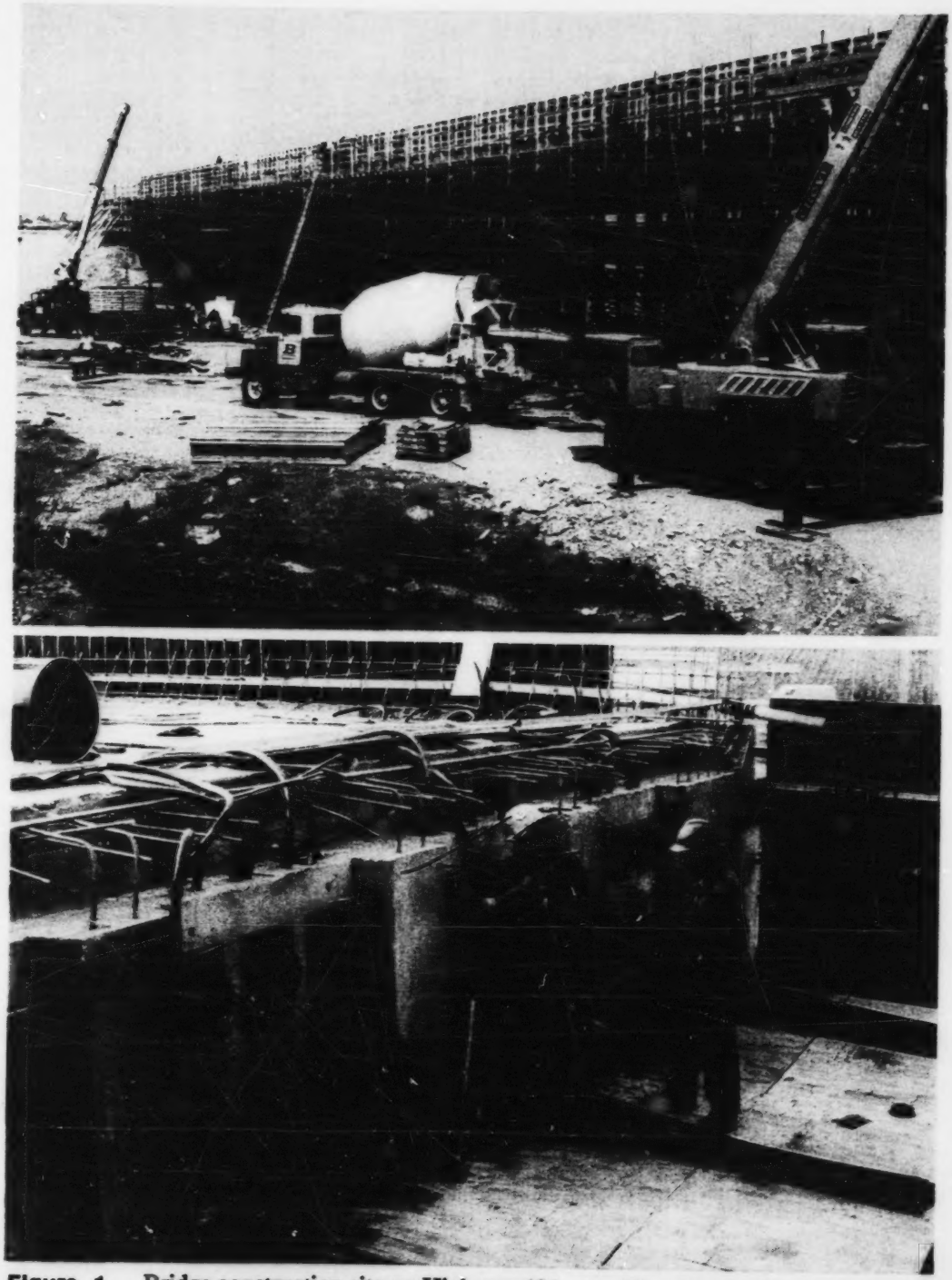


Figure 1. Bridge construction site on Highway 401, near Woodstock, Ontario *(upper)* and view of longitudinal tendons prior to grouting *(lower)*

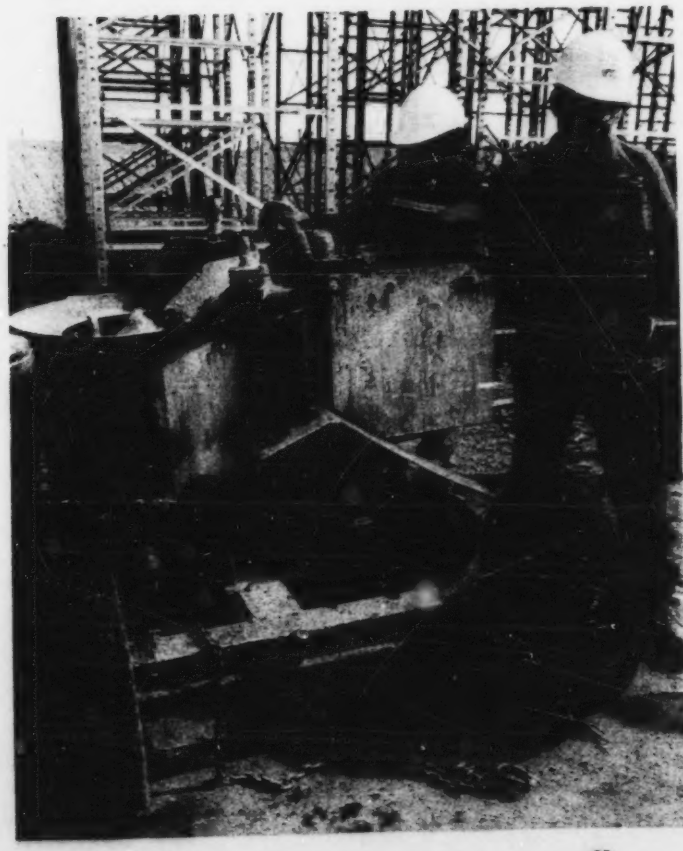


Figure 2. High-shear mixer and grout pump

Figure 3. Filling transverse tendon ducts with grout

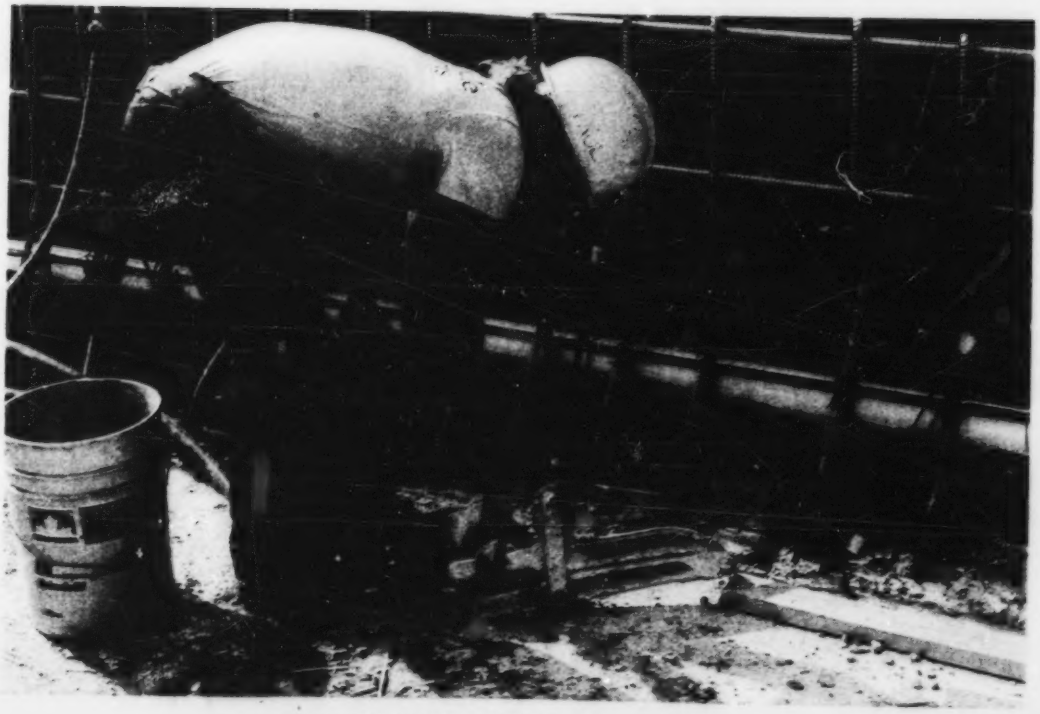


Figure 4. Sealing off plastic riser tubes

Figure 5. Cowles Model 1VG high-shear mixer used during preparation of cement grouts in the laboratory (see text)

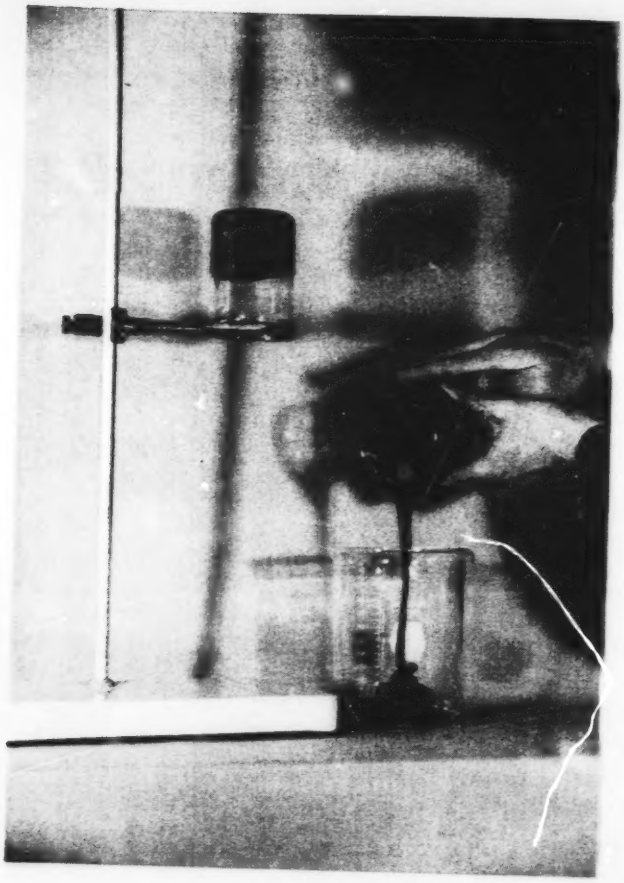


Figure 6. Thixotropic behaviour of PPC grout: 1 min after mixing, inverted beaker shows no tendency to flow *(left)*; gently tapping side of container promotes flow *(right)*

Figure 7. Measurement of relative viscosity using a Brookfield RVT Viscometer

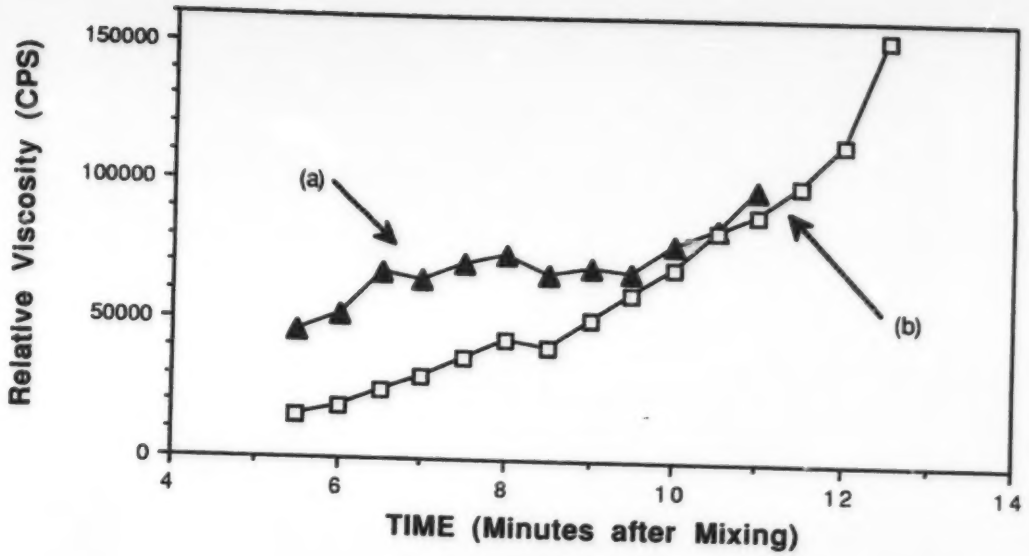


Figure 8. Variation of relative viscosity vs. time at 22°C for (a) Control PPC grout, and (b) PPC grout

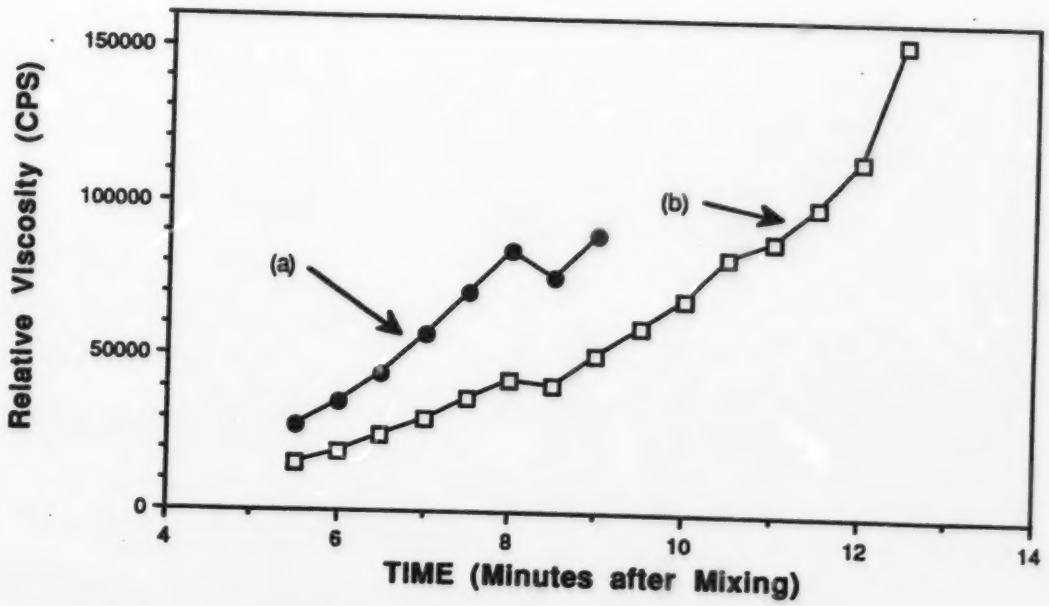


Figure 9. Variation of relative viscosity vs. time for (a) PPC grout at 30°C, and (b) PPC grout at 22°C.

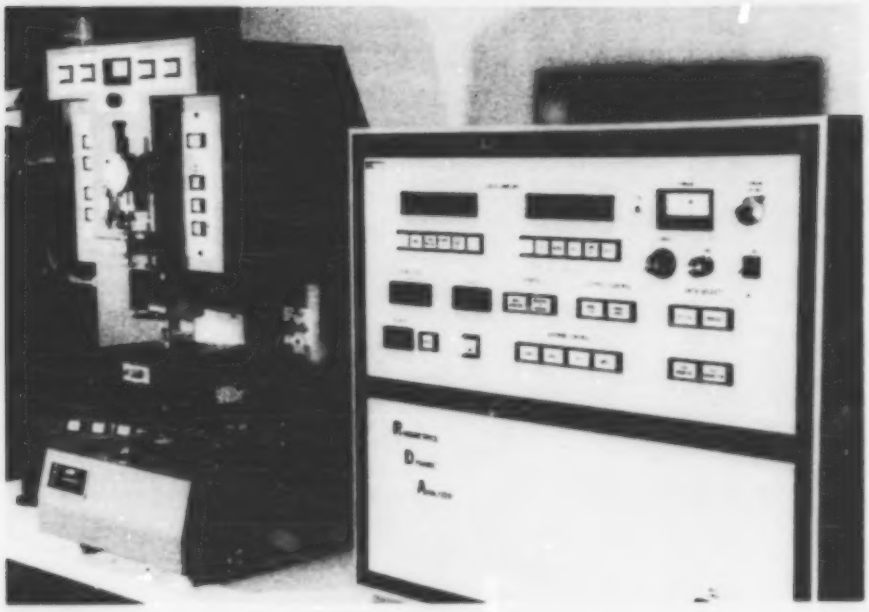


Figure 10. Rheometrics RDA-700 mechanical spectrometer used for dynamic mechanical analysis of grout

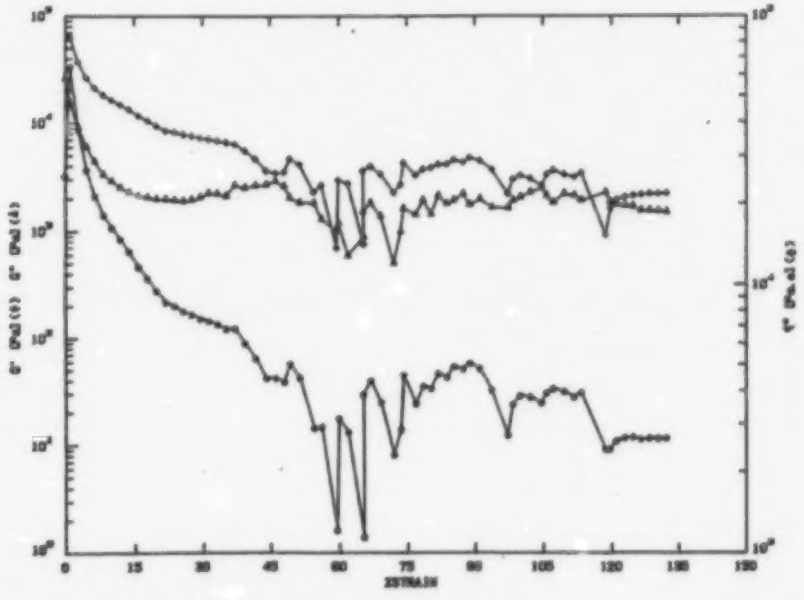


Figure 11. Strain sweep of PPC grout at 23°C (start of run 7 min after mixing) showing yield point at ~60% strain

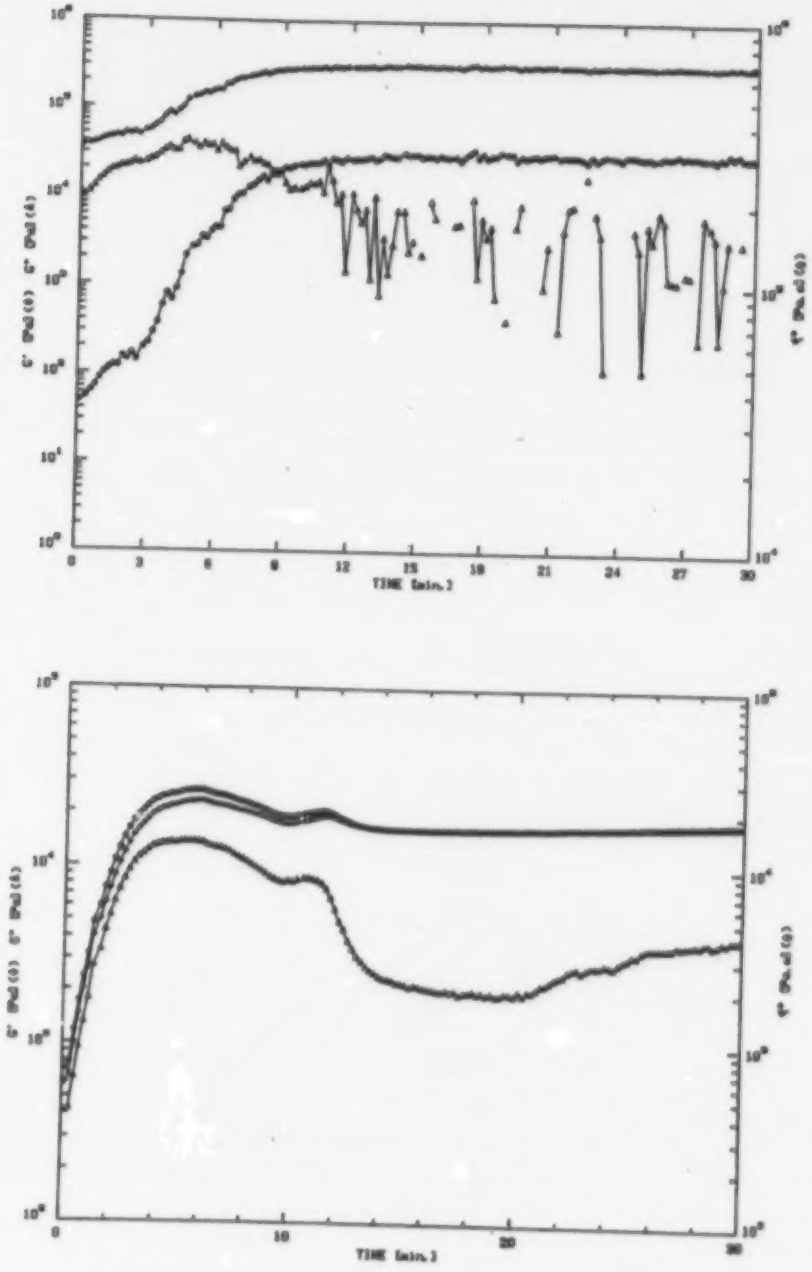


Figure 12. Time sweeps of PPC grout at 23°C at 1% strain *(upper plot)*, and 20% strain *(lower plot)* (start of run, 5 min from time of mixing)

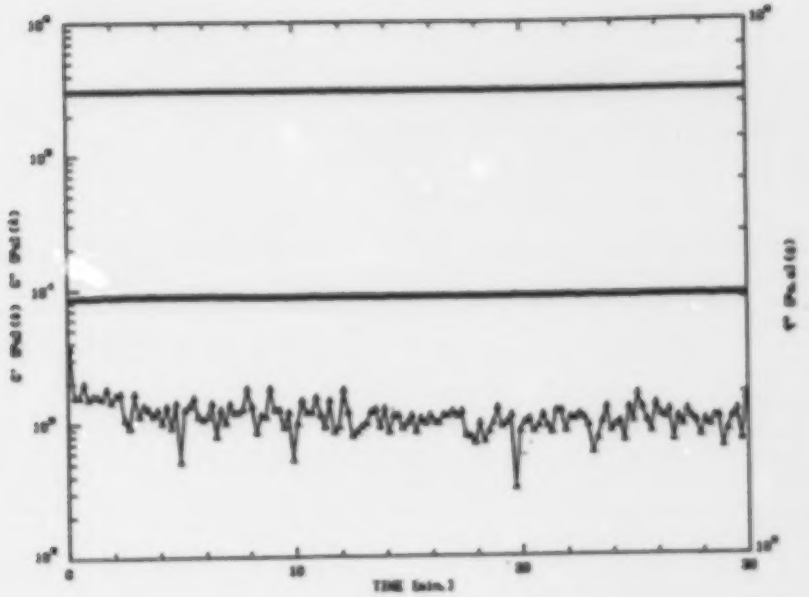


Figure 13. Time sweep of grout at 35°C and 20% strain (8 min after mixing)

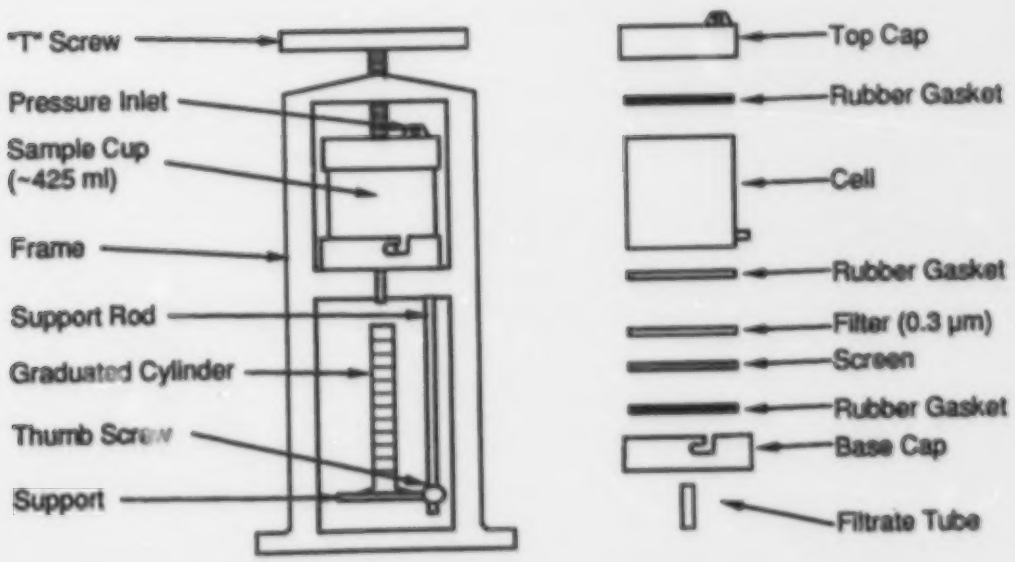


Figure 14. Filter press (NL Baroid/NL Industries) used to measure pressure induced fluid loss (i.e., water retentivity) in grouts

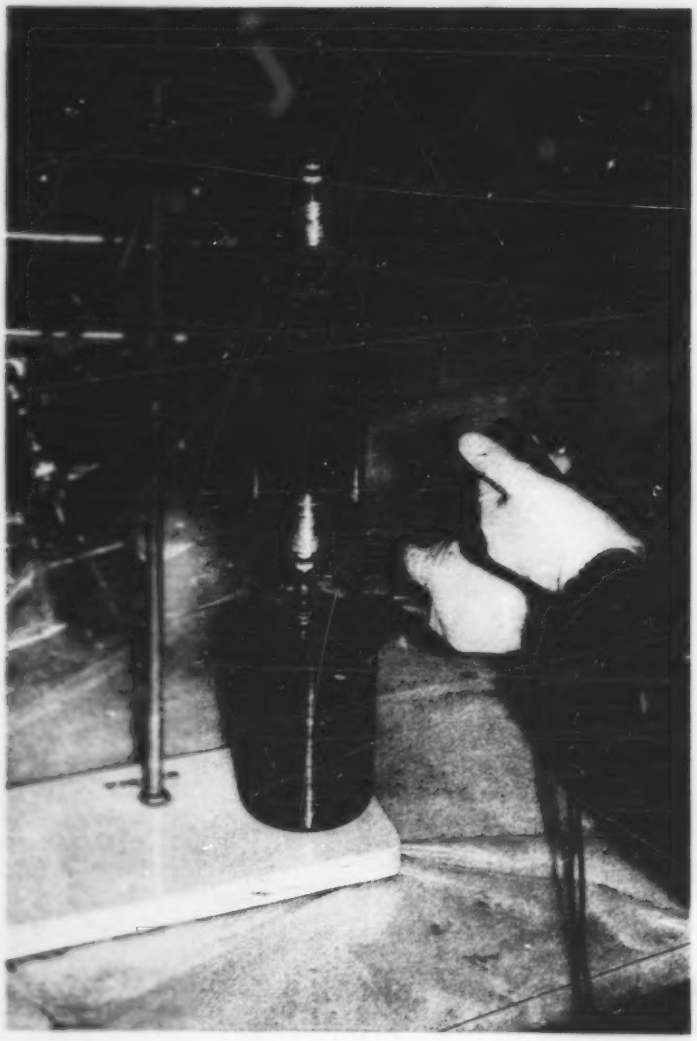


Figure 15. Cell used for measurement of efflux rate of grout under pressure

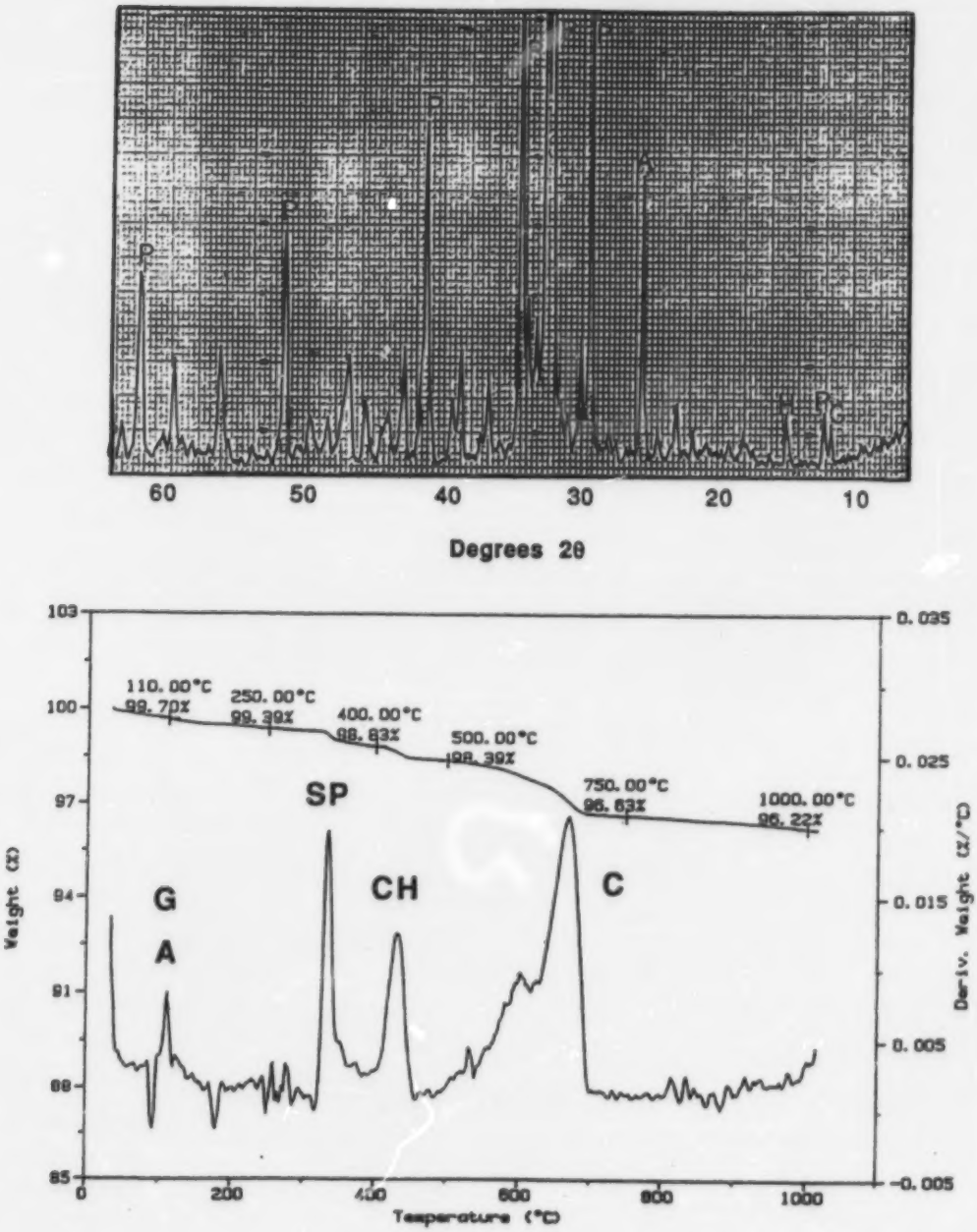


Figure 16. XRD *(upper)* and TGA *(lower)* traces for as-received (unhydrated) PPC grout. Phases identified as follows: **P**, unhydrated Portland cement, **E**, ettringite; **A**, anhydrite; **G**, gypsum; **CH**, calcium hydroxide; **C**, calcium carbonate; **SP**, superplasticizer

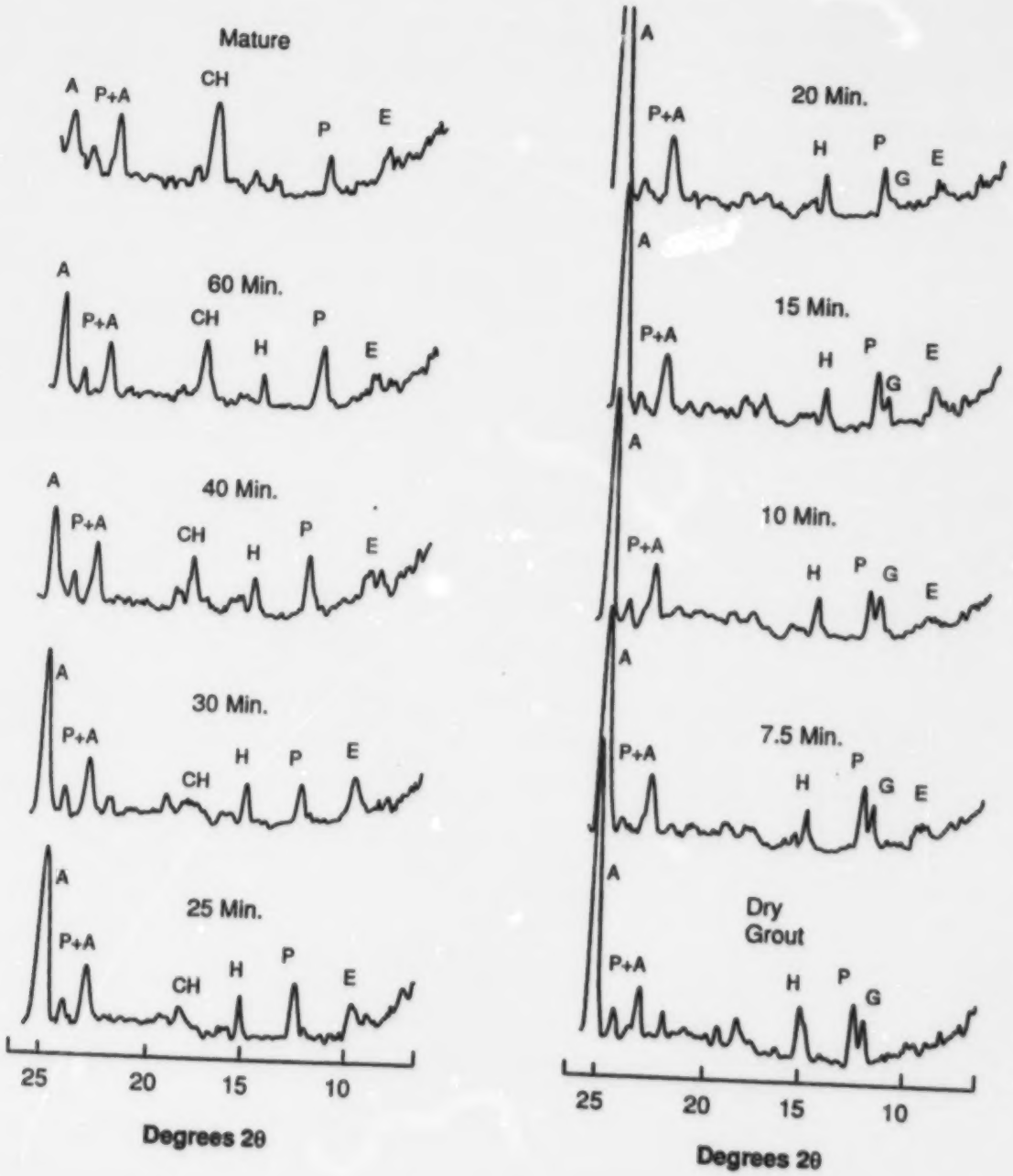


Figure 17. XRD patterns showing progressive hydration of PPC grout during 1-h period following mixing. Phases identified as follows: **P**, unhydrated Portland cement, **E**, ettringite; **G**, gypsum; **A**, anhydrite; **H**, calcium sulphate hemihydrate; **CH**, calcium hydroxide.

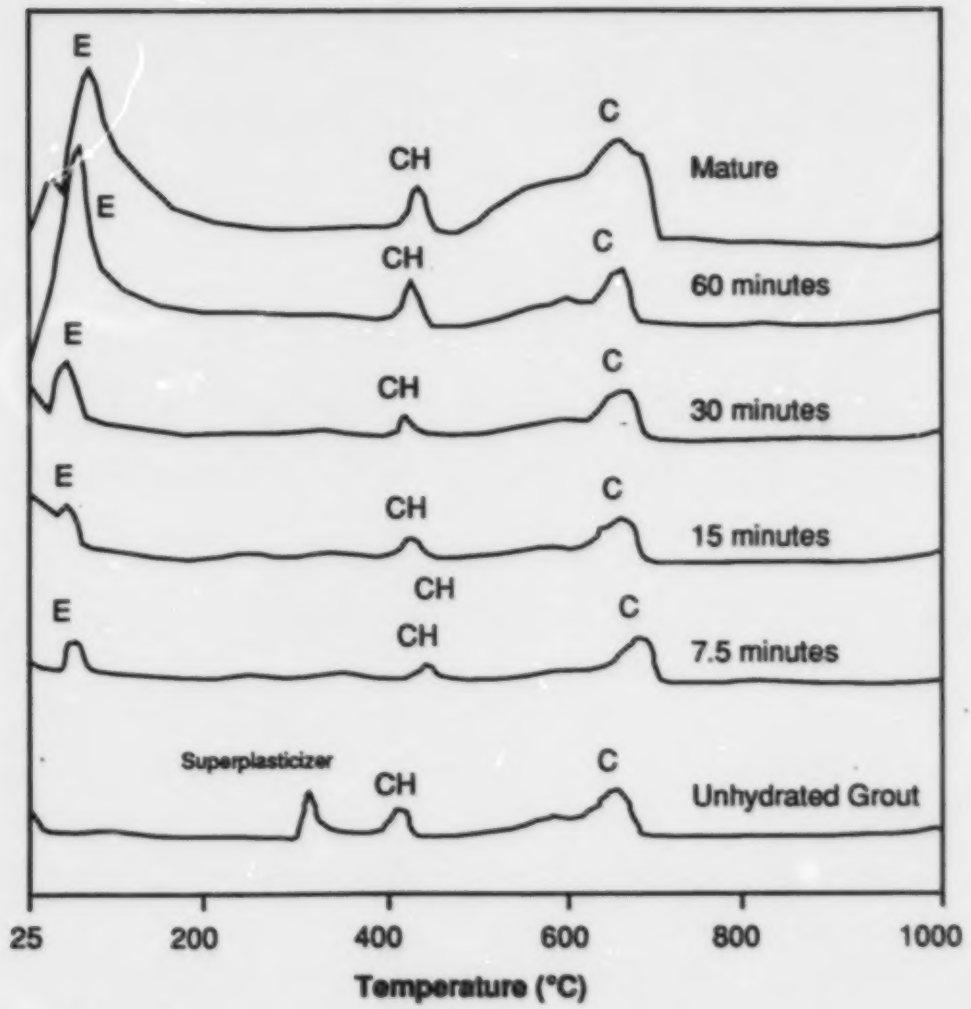


Figure 18. TGA curves showing progressive hydration of PPC grout during 1-h period following mixing, with reference to the unhydrated and mature grout. Phases identified as follows: **E**, ettringite; **CH**, calcium hydroxide; **C**, calcium carbonate

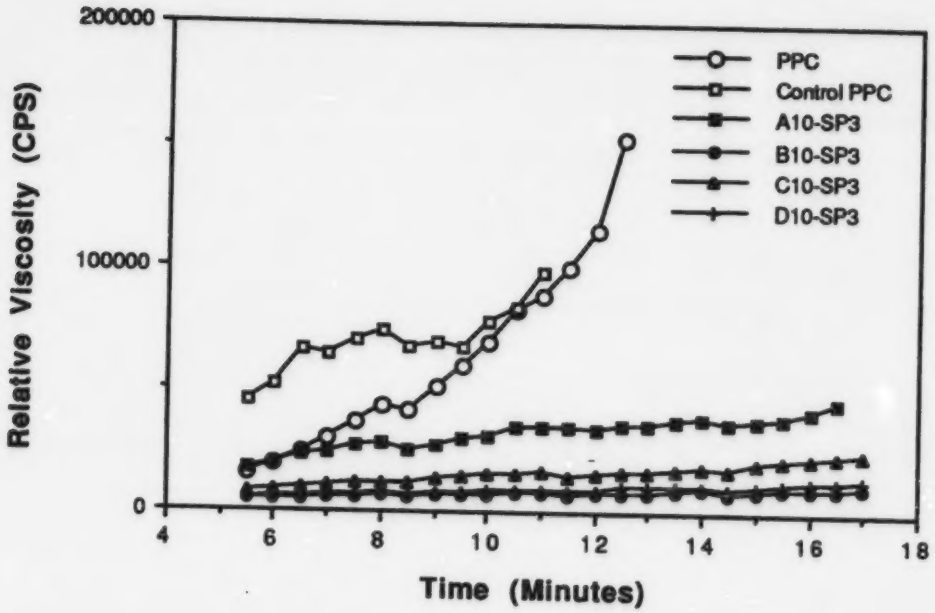


Figure 19. Effect of cement type on the relative viscosity of grout mixtures

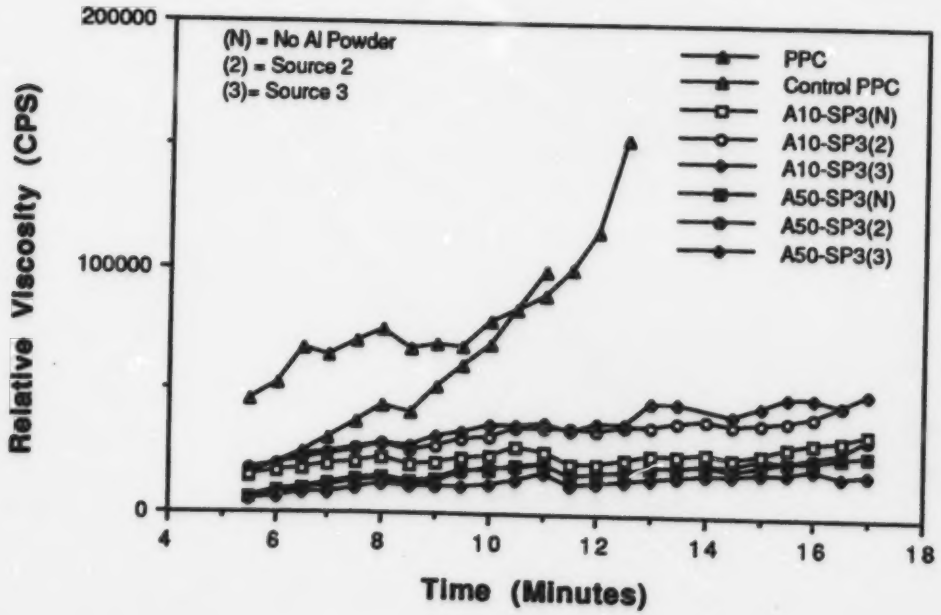


Figure 20. Effect of aluminum powder on the relative viscosity of grout mixtures

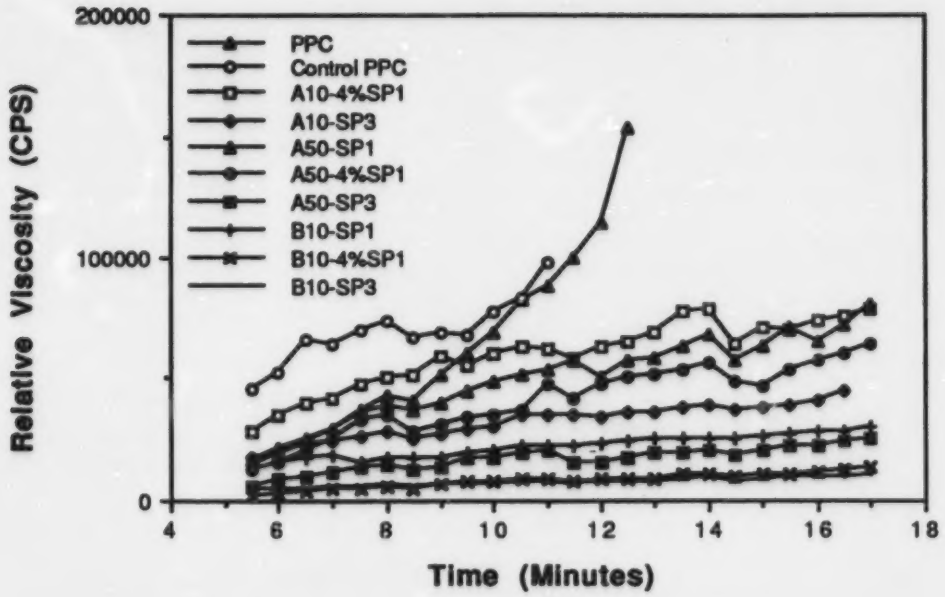


Figure 21. Effect of superplasticizer type on the relative viscosity of grout mixtures

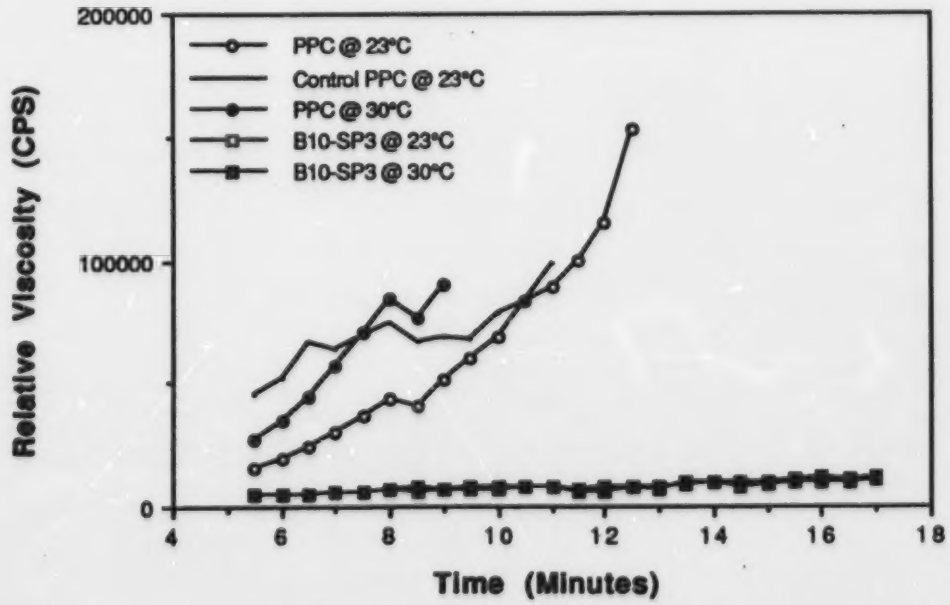


Figure 22. Effect of temperature on the relative viscosity of the PPC grout and the B10-SP3 grout

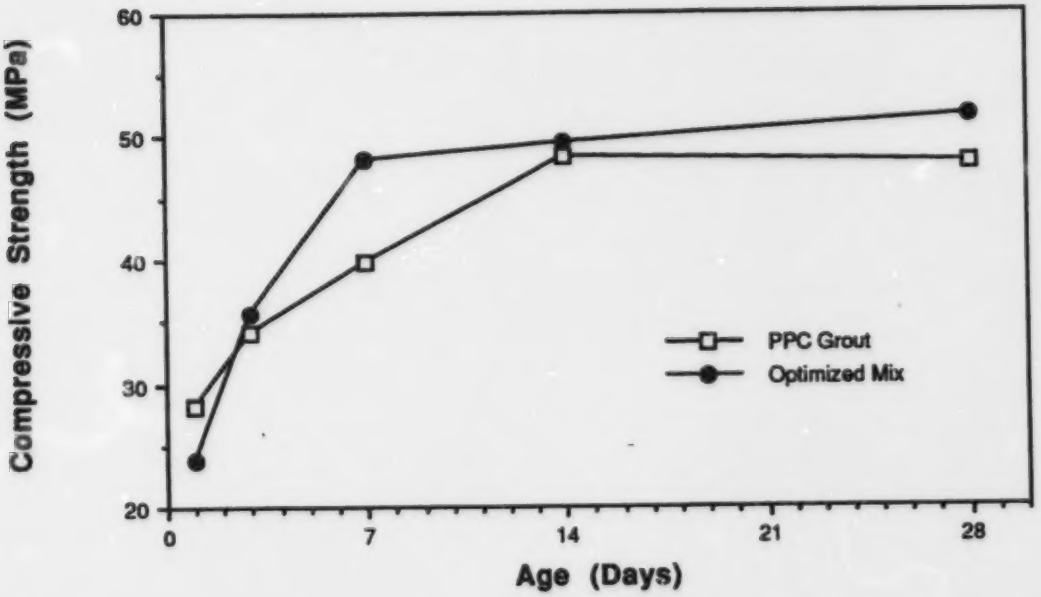


Figure 23. Compressive strength development of the PPC grout and the optimized grout

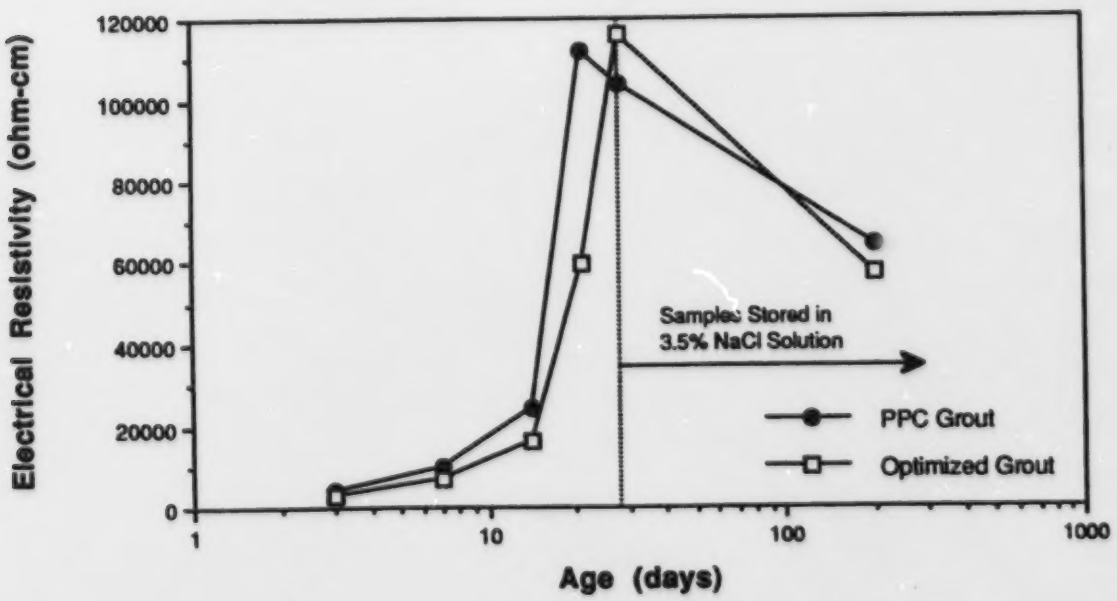


Figure 24. Comparison of the electrical resistivity of the PPC grout and the optimized grout